



**Draft**

**Work Plan  
Remedial Investigation  
Installation Restoration Program Site 6**

**Defense Fuel Support Point San Pedro  
San Pedro, California**

**August 2015  
Document Control No: KCH-2622-0100-0009**

Contract No: N62473-09-D-2622

CTO No: 0100

Prepared for:



Prepared by:





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**Department of the Navy**  
**Naval Facilities Engineering Command**  
**Southwest**

Prepared by



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# Acronyms and Abbreviations

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APP	accident prevention plan
BAMP	Biological Avoidance and Minimization Plan
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COPC	chemical of potential concern
DFSP	Defense Fuel Support Point
DoD	United States Department of Defense
DOT	United States Department of Transportation
DTSC	California Department of Toxic Substances Control
DWR	California Department of Water Resources
ELAP	Environmental Laboratory Accreditation Program
ERA	ecological risk assessment
FS	feasibility study
HHRA	human health risk assessment
HSA	hollow-stem auger
IDW	investigation-derived waste
IRP	Installation Restoration Program
JP	jet propellant
KCH	CH2M HILL Kleinfelder, A Joint Venture
msl	mean sea level
NAVFAC	Naval Facilities Engineering Command
Navy	United States Department of the Navy
NTU	nephelometric turbidity unit
OCP	organochlorine pesticide
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PID	photoionization detector
ppm	parts per million
PVB	Palos Verdes Blue Butterfly

PVC	polyvinyl chloride
RI	remedial investigation
RWQCB	California Regional Water Quality Control Board
SAP	sampling and analysis plan
SI	site inspection
SIM	selective ion monitoring
SSHIP	site safety and health plan
SVOC	semivolatile organic compound
TPH	total petroleum hydrocarbons
TPH-e	total petroleum hydrocarbons-extractable
TPH-p	total petroleum hydrocarbons-purgeable
USEPA	United States Environmental Protection Agency
UST	underground storage tank
VOC	volatile organic compound
WRD	Water Replenishment District

# 1.0 Introduction

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CH2M HILL Kleinfelder, A Joint Venture (KCH), has prepared this work plan to perform a remedial investigation (RI) at Installation Restoration Program (IRP) Site 6 at Defense Fuel Support Point (DFSP) San Pedro, California. DFSP San Pedro is shown on Figure 1-1, and IRP Site 6 is shown on Figure 1-2. KCH is performing this work for the United States Department of the Navy (Navy), Naval Facilities Engineering Command (NAVFAC) Southwest Division in accordance with Contract No. N62473-09-D-2622, Contract Task Order No. 0100.

IRP Site 6 is a former disposal area referred to as the South Ravine, shown on Figure 1-2. A site inspection (SI) was finalized at IRP Site 6 in 1993 (Jacobs, 1993). The SI recommended that an RI/feasibility study (FS) be conducted at IRP Site 6 based on the presence of semivolatile organic compounds (SVOCs) and metals in soil at concentrations above applicable screening criteria. The goal of the RI is to answer the following questions:

- Is the nature and extent of debris at IRP Site 6 adequately characterized?
- Are chemicals at IRP Site 6 adequately delineated?
- Do concentrations of chemicals in soil and groundwater pose an unacceptable risk to human health or the environment?

In order to achieve the goal of the RI, soil and groundwater sampling will be conducted, the conceptual site model identifying receptors and pathways will be created and refined, and a human health risk assessment (HHRA) and ecological risk assessment (ERA) will be completed. The HHRA and ERA will be completed after all analytical data are collected.

KCH will subcontract services for subsurface utility locating, geophysical survey, biological survey, hollow-stem auger (HSA) drilling, trenching, investigation-derived waste (IDW) disposal, location surveying, chemical analysis, and data validation.

The results of the field investigation activities will be reported by KCH in an RI report. The following are the main objectives for the RI report:

- Summarize site background, historical data, and environmental settings.
- Present the data from field investigations and assess current site conditions, including nature and extent of contamination.
- Evaluate the fate and transport of site contaminants to assess their potential to migrate and affect human or ecological receptors.
- Present human health and ecological risk assessments for IRP Site 6.

Appendix A includes the sampling and analysis plan (SAP) for planned investigation activities at IRP Site 6. Appendix B includes the Biological Avoidance and Minimization Plan (BAMP). This work plan also references the accident prevention plan (APP) and site safety and health plan (SSHP), which are being submitted separately.

## 1.1 Project Schedule

Preparatory activities for field sampling are scheduled to begin in August 2015, and the field sampling is scheduled for October 2015. The expected completion date for the RI report is March 2016. The anticipated schedule for fieldwork and reporting is presented in Worksheet #16 of the SAP (Appendix A).

## 1.2 Project Organization and Points of Contact

The individuals listed in the following chart are primary points of contact for this work plan:

Name	Title/Role	Organization	Telephone Number	Email Address or Mailing Address
Brenda Reese	Remedial Project Manager	NAVFAC Southwest	(619) 532-4209	brenda.reese@navy.mil
Alan Hsu	Lead Remedial Project Manager	California DTSC	(714) 484-5395	ahsu@dtsc.ca.gov
Eric Johansen	Task Order Manager	KCH	(619) 831-4605	ejohansen@kleinfelder.com

Note:  
DTSC = Department of Toxic Substances Control

Worksheets #3 and #4 of the SAP (Appendix A) show complete project roles and contact information for key Navy, KCH, subcontractor, and regulatory agency individuals involved with the proposed work at IRP Site 6. A project organization chart is provided in Worksheet #5 of the SAP (Appendix A).

## 1.3 Work Plan Organization

This work plan is organized as follows:

**Section 1.0, Introduction** – An overview of the site, project schedule, project organization, project points of contact, and the organization of the work plan.

**Section 2.0, Site Conditions and Background** – A summary of the DFSP San Pedro and IRP Site 6 site background, geology, hydrogeology, climate, ecological setting, site description, and previous investigations conducted to date.

**Section 3.0, Regulatory Framework** – A summary of decision makers, technical or regulatory standards, and permitting requirements.

**Section 4.0, Proposed Scope of Work** – A description of the planned RI activities and a statement that safety and health during RI activities will be performed in accordance with the APP and SSHP.

**Section 5.0, Reporting** – A description of the report that will be prepared to document results of the investigation.

**Section 6.0, References** – A list of documents cited in this work plan.

**Appendix A** – SAP for RI at IRP Site 6.

**Appendix B** – BAMP for RI at IRP Site 6.

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## 2.0 Site Conditions and Background

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DFSP San Pedro is located in the community of San Pedro, in the city and county of Los Angeles, California, east of the city of Rolling Hills Estates and west of the city of Long Beach. DFSP San Pedro occupies approximately 331 acres. DFSP San Pedro is shown on Figure 1-1. It is bordered on the north and south by residential and commercial property, on the east by the ConocoPhillips oil refinery, and on the west by Green Hills Memorial Park (Figure 1-2). The facility became operational in 1943, with the primary mission of storage and distribution of fuels to support military operations in California, Arizona, and Nevada. The majority of the underground storage tanks (USTs) and associated fuel pipelines were installed in 1944. Additional operational activities included storage of small arms ammunition after World War II and construction of a small arms pistol range, housing areas, and baseball fields. The Navy operated the facility until 1980, when operational responsibility was transferred to the Defense Energy Support Center, a branch of the Defense Logistics Agency.

### 2.1 DFSP San Pedro Background

DFSP's mission involves truck and pipeline transport of fuel to United States military units.

### 2.2 Geology and Hydrogeology

The geology and hydrogeology information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013), which is approximately 100 feet east of IRP Site 6.

#### 2.2.1 Regional Geologic Setting

The DFSP facility is near the northern end of the Peninsular Ranges geomorphic province. The Santa Ana, San Jacinto, and Laguna Mountains are included in this region. These mountains are composed of mostly Cretaceous Period (65 to 145 million years old) igneous rocks and Tertiary Period (2.6 million to 65 million years old) marine sedimentary rocks (Jacobs, 1993). The Peninsular Range geomorphic province is characterized by northwest-trending mountain ranges and valleys bounded by right-lateral strike-slip faults. The primary geologic faults include the Palos Verdes, Newport-Inglewood, Whittier-Elsinore, and San Jacinto. DFSP San Pedro is at the southwestern tip of the Los Angeles basin. The Palos Verdes Hills, rising to an elevation of about 1,300 feet, are the most prominent feature in the area.

The Palos Verdes fault extends from the offshore area of the San Pedro Shelf into the Los Angeles harbor area and then along the northeastern margin of the Palos Verdes Hills to the Redondo Beach area. The fault plane is steeply dipping to the west offshore and exhibits right lateral strike-slip motion. Onshore, the fault has uplifted the Palos Verdes Hills and thrust them over the Torrance Plain along a steep fault plane dipping 65° to 70° to the southwest (Schell, 2008). The exact onshore location of the Palos Verdes fault is equivocal. The main trace of the fault zone has been mapped as passing through the northern portion

of DFSP San Pedro (Bechtel, 1995). Folding of the local rocks has resulted in northwest-trending anticlines and synclines. The Gaffey anticline trends through the DFSP facility, with the axis about 500 feet north of IRP Site 6 (California Geological Survey, 2003). Pleistocene history of movement of the Palos Verdes fault system is recorded by the presence of 13 distinct wave-cut marine terraces along the coastal front (Woodring et al., 1946). The marine terraces are wave-cut surfaces incised and successively uplifted to their present elevations. The facility is on Marine Terrace No. 1, the terrace with the lowest altitude in the sequence of 13 terraces.

Elevations across DFSP San Pedro vary from approximately 25 feet above mean sea level (msl) on the eastern side of the facility, to approximately 260 feet above msl in the west (SGI, 2009). The hills are cut by several ravines and gullies. The primary ravine (usually referred to as the Central Ravine) bisects the facility and trends east-west.

The DFSP facility is underlain by metamorphic basement of late Jurassic- to late Cretaceous-age and sedimentary deposits ranging from Miocene to Holocene. The stratigraphy is characterized from youngest to oldest as follows (Bechtel, 1995):

- **Fill.** Manmade fill deposits are present throughout the facility. Most of these deposits are uncontrolled fill materials placed in former ravines, such as IRP Site 6. These materials consist primarily of loose to dense silty sand and clayey sand mixed with debris and construction rubble.
- **Alluvium.** Relatively thin deposits of Holocene alluvium are present in existing ravines and at depths along previously buried ravines. These soils are generally loose- to medium-dense, dark brown sand and silty sand.
- **Terrace Deposits (Lakewood Formation).** Late Pleistocene marine and nonmarine terrace deposits are locally found in the facility. These undifferentiated deposits vary from 0 to 700 feet thick. The lower portion has been reported to consist of fairly continuous dense to very dense sand and gravel with finer-grained lenses, while the upper portion consists of predominantly fine-grained flood plain deposits with discontinuous lenses of variable lithologies (Wilkinson, 2003). These deposits have been grouped together and mapped in the area as the Lakewood Formation (Wilkinson, 2003; OHM Remediation Services Corp., 2001).
- **San Pedro Formation.** The Pleistocene (approximately 11,000 to 1.6 million years old) San Pedro Formation unconformably overlies the Monterey Formation. The San Pedro Formation generally consists of marine gravels, silty sands, and clays in stratified layers and some soft limestone. Bivalve shells are locally present in San Pedro Formation sediments. San Pedro Formation appears at the ground surface across the DFSP property.
- **Monterey Formation.** The Monterey Formation unconformably overlies the metamorphic basement. Monterey Formation is middle to upper Miocene (5 million to about 15 million years old), composed of three sedimentary members of marine origin: Altamira Shale, Valmonte Diatomite, and Malaga Mudstone. Malaga Mudstone is the uppermost member and is the only unit that has been encountered at DFSP San Pedro. This unit is exposed along the sidewalls of the upper Central Ravine and has been encountered at depth at DFSP San Pedro. Malaga Mudstone has been characterized in

soil boring logs as a clayey siltstone with some thin sandstone interbeds and appears olive-green and weathered (Jacobs, 1993).

- **Metamorphic Basement.** Late Jurassic to late Cretaceous-age (approximately 65 to 135 million years old) metamorphic rocks of the Franciscan Formation (Catalina schist facies), typically hard, layered, and composed primarily of schist. The Franciscan Formation generally lies 1,200 to 4,000 feet beneath the Palos Verdes Hills.

## 2.2.2 Regional Hydrology and Hydrogeology

The regional surface drainage flows west to east from the Palos Verdes Hills, across the facility and into Los Angeles Harbor. Local surface water runoff is generally controlled by drainage through existing ravines, overland flow, and engineered drainage devices (Bechtel, 1995).

Three bodies of surface water are located near DFSP San Pedro: Harbor Lake, Palos Verdes Reservoir, and Los Angeles Harbor. Harbor Lake, formerly known as Bixby Slough, is located 0.25 mile northeast of DFSP San Pedro. Harbor Lake is a protected wildlife refuge and is part of the local flood control system. The Palos Verdes Reservoir is located approximately 0.5 mile west of the facility. The reservoir is a covered treated-water storage facility for water supply. The California Water Service Company provides water from the reservoir to the Palos Verdes area, and the Los Angeles Department of Water and Power withdraws water from the reservoir to service parts of San Pedro. Los Angeles Harbor, located approximately 0.9 mile east of the site, is bordered by heavy industrial facilities. The land surface in this area has been altered to facilitate industrial development. Los Angeles Harbor is used as a main corridor for military, commercial, and privately owned ocean-going vessels and commercial fishing operations.

Saltwater intrusion barriers are maintained along the Los Angeles and Orange County sections of the coastal plain. In Los Angeles County, imported and recycled water is injected to maintain a saltwater intrusion barrier (DWR, 2003).

California Department of Water Resources (DWR) identified the three major aquifers in the West Coast Basin: the Silverado and Lynwood aquifers, part of the San Pedro Formation, and the Gage aquifer, part of the Holocene and latest Pleistocene deposits (DWR, 1961; Wilkinson, 2003). The Gage aquifer is the lowermost aquifer of the Lakewood Formation and is of upper Pleistocene age. The Lynwood and Silverado aquifers are lower Pleistocene and are the uppermost and lowermost aquifers in the San Pedro Formation, respectively. The San Pedro Formation contains the most extensive aquifers in the West Coast Basin and is the principal source of groundwater in the basin (Jacobs, 1993). The marine and continental deposits of the Lakewood Formation include the Gage aquifer, consisting of fine sand with interbedded silty and sandy clay, and silty and clayey fine- to medium-grained sand. The Gage aquifer has been reported to be approximately 100 feet thick near DFSP San Pedro (OHM, 2001). The hydrogeologic units used by DWR, (DWR, 1961) that are well defined in the interior of the basin are less clear in the vicinity of DFSP San Pedro and the neighboring ConocoPhillips refinery (Wilkinson, 2003). For the purposes of the hydrogeologic evaluation and discussion, five hydrostratigraphic units have been defined for use at the neighboring ConocoPhillips Refinery (Wilkinson, 2003). The upper three units

correlate to the Lakewood and upper San Pedro Formations and are believed to be representative of the Gage aquifer.

Depth to first-encountered groundwater is variable throughout the DFSP, ranging from about 10 to 35 feet bgs near the pump house, to approximately 15 to 134 feet bgs at the South Tank Farm area, and 43 to 74 feet bgs in the administrative area (Figure 1-2; SGI, 2008). The groundwater flow direction of the uppermost hydrogeologic unit is generally to the south, based on a recent groundwater monitoring event (SGI, 2014).

The California Regional Water Quality Control Board (RWQCB) adopted a basin plan (1994) that designates groundwater beneath DFSP San Pedro for municipal and domestic beneficial use. Groundwater at DFSP San Pedro is not currently used for any municipal or industrial purposes (no water production wells are present at the facility). The database of the Water Replenishment District (WRD) of Southern California indicates the presence of seven active supply wells within an approximately 4-mile radius of IRP Site 6 (WRD, 2009). The closest supply well is approximately 1 mile away at the ConocoPhillips refinery and is screened in the Silverado aquifer (Trihydro, 2008).

## 2.3 Climate

The climate information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013).

The climate in the southern California coastal region is semiarid and moderate. Seasonal variations are mild. Low clouds are predominant in the region in the early morning and at night. The most dramatic weather occurs during the winter, when patterns of rainstorms and Santa Ana winds (hot, dry winds that blow from the east) are possible. Based on climate records for the area, temperatures range from an average low of 44 degrees Fahrenheit (°F) in January to an average high of 79 °F in August. Average monthly precipitation ranges from 0.02 inch in July to 3.2 inches in February, with an average annual precipitation of 13.5 inches (Western Regional Climate Center, 2010).

## 2.4 Ecological Setting

The ecological setting information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013).

Vegetation at IRP Site 6 is anticipated to be similar to vegetation at IRP Site 32, which consists of chaparral and grassland species, including mustard (*Hirschfeldia incana*), thistle (*Centaurea melitensis*), and various wild grasses such as wild oat (*Avena fatua*). Previous reports included inventories of plants and animals found in and around the DFSP (Jacobs, 1993; OHM Remedial Services Corp., 2001), but surveys specific to IRP Site 6 were not performed.

Potential bird species at IRP Site 6 include the common raven (*Corvus corax*), American kestrel (*Falco sparverius*), American robin (*Turdus migratorius*), California towhee (*Pipilo crissalis*), European starling (*Sturnus vulgaris*), house finch (*Carpodacus mexicanus*), mourning dove (*Zenaidura macroura*), rock dove (*Columba livia*), and western meadowlark (*Sternella neglecta*).

Mammals potentially present at IRP Site 6 include species of small rodents and larger mammals. Rodents include the California ground squirrel (*Spermophilus beecheyi*), deer mouse (*Peromyscus maniculatis*), dusky-footed woodrat (*Neotoma fuscipes*), fox squirrel (*Sciurus niger*), and western harvest mouse (*Reithrodontomys megalotis*). Larger mammals include the badger (*Taxidea taxus*), black-tailed jackrabbit (*Lepus californicus*), coyote (*Canis latrans*), gray fox (*Urocyon cinereoargenteus*), raccoon (*Procyon lotor*), red fox (*Vulpes vulpes*), and striped skunk (*Mephitis mephitis*).

A number of special status species could occur at IRP Site 6, including the federally listed threatened Coastal California gnatcatcher (*Polioptila californica californica*) and the federally listed endangered Palos Verdes Blue Butterfly (PVB) (*Glaucopsyche lygdamus palosverdensis*). The PVB was believed to be extinct for 11 years until discovered at DFSP San Pedro on March 10, 1994 (Mattoni, 1994). The PVB is closely associated with its larval food plants, California locoweed (*Astragalus trichopodes* var. *lonchus*) and common deerweed (*Lotus scoparius*). The PVB completes its entire life cycle near its host plant. Deerweed was observed at IRP Site 6 during a June 6, 2014 site walk.

## 2.5 IRP Site 6 Description

IRP Site 6 was defined after a 1990 site visit identified the South Ravine as a former disposal area. Paint spills, rusted 55-gallon drums, and 1- and 5-gallon cans containing varying amounts of unidentified liquids were noted. Wooden boards and furniture, brush, metal pipe, concrete, and tires were also visible during the site visits on April 9, 2014 and June 6, 2014.

The South Ravine has northwestern and northeastern branches as shown on Figure 2-1. The northwestern branch has been filled almost to grade. During the SI, a fill depth of 19 feet bgs was identified in this branch. The northeastern branch was identified as being incised (narrower and with steeper sides), with fill visible in this area as well (Jacobs, 1993).

Engineering of the south-sloping ravine bottom in IRP Site 6 is apparent in a 1943 topographic map (United States Navy Fuel Depot, 1943). A concrete v-drain was present during a 1990 site visit in the southern end of the South Ravine bottom. Sands present in this area may be from the upper part of the ravine where vertically cut banks are visible.

The history of disposal in the South Ravine is unknown. Base personnel did not know of filling operations, so it is likely that disposal at this site was unscheduled (Jacobs, 1993).

## 2.6 Summary of Previous Investigations

A fuel spill occurred in 1981 or 1982 at a nearby tank. An estimated 10,000 gallons of diesel fuel reportedly flowed east into the southern end of the South Ravine. No cleanup of the spill was attempted. Two borings (WCB-10 and WCB-11, shown on Figure 2-1) were placed by Woodward-Clyde Consultants in the probable path of the spill. Soil samples collected from 2 to 15 feet bgs from WCB-10 and 2 to 4 feet bgs from WCB-11 were analyzed for total petroleum hydrocarbons (TPH) as gasoline, kerosene, diesel, jet propellant (JP)-4, and unknown hydrocarbons. There were no concentrations above the detection limit of

10 milligrams per kilogram for the four samples submitted for laboratory analysis (Jacobs, 1993).

An SI was conducted at IRP Site 6 in 1992. IRP Site 6 consists of a main north-south trending drainage with a small northwest-southeast trending tributary ravine (i.e., the northwestern branch of the South Ravine), shown on Figure 2-1. During the SI, along the surface of the main drainage, miscellaneous debris, including metal, glass, and wood fragments, were present. The upper portions of the tributary drainage were covered with an asphalt-like substance, possibly the remnants of a fuel spill from Tank 5 (Jacobs, 1993).

Six soil borings (B01 through B06) were installed during this investigation. Attachment 1 of Appendix A contains the locations of historical soil borings and a summary of the analytical data collected during the SI (Jacobs, 1993). During the boring installations, it was noted that the main portion of the South Ravine and tributary were filled with approximately 10 to 20 feet of assorted fill debris. Abundant broken glass fragments were encountered at boring B01 in the northern end of the South Ravine. At boring B05, volatile organic compounds (VOCs) (benzene, ethylbenzene, toluene, xylenes, and various ketones) and SVOCs (primarily polycyclic aromatic hydrocarbons [PAHs]) were detected in soil. Although the soil was logged as fill material to a depth of 14 feet bgs, the lower portions of this interval may be alluvium with abundant residual fuel products. The soils were overlaid with silty soil fill materials.

The main South Ravine axis was also capped with a granular fill sand that was apparently emplaced during installation of a concrete v-drain. The v-drain is confined to the main South Ravine axis. At the southern end of the South Ravine, rip-rap is present along the v-drain in the vicinity of boring B06. No fill soils were encountered in boring B06. Boring B07 was drilled east of the northern part of IRP Site 6. Clean sands of the San Pedro Formation were encountered in this boring (Jacobs, 1993).

The sampling at IRP Site 6 during the SI indicated that elevated levels of chemicals of potential concern (COPCs) were present in soil, particularly in the northwestern portion of the ravine. COPCs identified in soil during the SI include fuels, organic lead, metals, SVOCs, and organochlorine pesticides (OCPs). Groundwater was not sampled during the SI. An RI/FS was recommended for IRP Site 6 based on these exceedances.

## 3.0 Regulatory Framework

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The following section presents the project regulatory framework.

### 3.1 Principal Decision Makers

Ongoing environmental restoration activities at DFSP San Pedro are overseen by the Navy. As the lead federal agency, the Navy has authority over evaluation of risk, remedy selection, and overall public participation at DFSP San Pedro. The Navy coordinates with DTSC, RWQCB, and the California Department of Fish and Wildlife through document development and reviews, remedial decisions, and remedial actions at IRP sites.

### 3.2 Technical or Regulatory Standards

The IRP Site 6 RI will be conducted under the procedural framework and schedule for the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The cleanup programs under CERCLA and the Superfund Amendments and Reauthorization Act include the Defense Environmental Restoration Program and the IRP activities performed in accordance with CERCLA.

### 3.3 Permitting Requirements

Los Angeles County Department of Public Health has permitting requirements for installation and destruction of groundwater monitoring wells. Well permit applications will be submitted for the proposed wells; however, the Navy does not pay permit fees for CERCLA actions such as the RI at IRP Site 6, in accordance with Section 121(e) of CERCLA 1980 (42 United States Code, Section 9621[e]), as amended. Groundwater monitoring well sampling does not require permits.

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## 4.0 Proposed Scope of Work

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The technical approach for the proposed scope of work, including sampling design and procedures, is presented in detail in Worksheets #14 and #17 of the SAP (Appendix A). This section discusses general field activities, preparatory activities, safety and health, and the scope of work proposed for IRP Site 6.

### 4.1 Preparatory Activities

Prior to beginning fieldwork, the following preparatory activities will take place:

- The Navy Remedial Project Manager will be notified about the anticipated work.
- Sampling personnel will review the appropriate sections of the SAP and BAMP (Appendices A and B) and sign the project sign-off sheet.
- Affected personnel will read the APP and associated SSHP and sign an acknowledgement form.
- Field personnel will obtain the appropriate access to the work areas from the Navy.
- Excavation permits for drilling and sampling activities will be obtained from NAVFAC Southwest Public Works at least 21 days prior to mobilization. Proposed boring locations will be marked in the field by KCH, prior to utility locating.
- Underground Services Alert will be notified at least 2 full working days in advance of drilling or subsurface sampling activity. Additionally, KCH will survey the site using geophysical methods to identify potential subsurface obstructions or utilities at drilling, trenching, and sampling locations. Proposed sampling locations will be modified as necessary to avoid utilities and subsurface obstructions.
- Mobilization activities will include site preparation, movement of equipment and materials to the site, and orientation of field personnel. Upon receipt of the required authorizations, site personnel will be mobilized to the site.

### 4.2 RI Field Activities

The RI at IRP Site 6 will be implemented using the approaches outlined in the following sections. A detailed description of the RI activities is presented in the SAP (Appendix A).

#### 4.2.1 Utility Locating/Geophysical Survey

A geophysical survey will be conducted at IRP Site 6 to assess the lateral extent of landfill debris to aid in the placement of sampling locations. This effort is necessary in the northern half of the site where debris are expected. Because of thick vegetation and steep terrain in the northeastern ravine, a traditional geophysical grid is not practical. Instead, geophysical transect lines will be used. The lines will consist of approximately 4,000 feet of linear

coverage with one transect parallel to each main ravine and several transects perpendicular across the ravines. Actual transect locations will be identified in the field, based on access considerations.

The geophysical subcontractor will also clear subsurface utilities at IRP Site 6 sample locations before drilling and trenching activities begin. All clearances needed for borehole drilling will be obtained in accordance with the Navy's established procedures and requirements. All locations will be marked in the field with a wooden stake, and the sample location will be indicated on a map.

## **4.2.2 Soil Sampling**

Soil sampling will be conducted at IRP Site 6 to address the following goals for the RI for IRP Site 6:

- Is the nature and extent of debris at the site adequately characterized?
- Are COPCs inadequately delineated?
- Do COPCs in soil pose an unacceptable risk to human or ecological receptors?

Soil samples will be collected via hand augering, HSA drilling, and trenching.

### **4.2.2.1 Hand Augering**

A hand auger will be used to clear each boring location down to approximately 5 feet bgs at IRP Site 6. The hand auger will also be used to collect soil samples from five soil borings to 10 feet bgs (see Figure 2-1). Soil samples will be collected from 0 to 0.5 foot bgs, 4 to 6 feet bgs, and 8 to 10 feet bgs. Soil sampling depth may be adjusted in the field based on changes in lithology, soil headspace photoionization detector (PID) readings, color, odor, and field geologist discretion. Analytical samples will be collected as explained in Section 4.2.2.4.

### **4.2.2.2 Hollow-Stem Auger Drilling**

Prior to drilling at IRP Site 6, each boring location will be cleared down to approximately 5 feet bgs with a decontaminated hand auger. Seven borings will be advanced by HSA drilling using a track-mounted HSA drill rig equipped with 8-inch-diameter augers. The locations of the borings are shown on Figure 2-1. Five of the borings (SB-1 through SB-05) will extend up to approximately 20 feet bgs, and two of the borings (MW-01 and MW-02) will extend up to approximately 150 feet bgs for installation of groundwater monitoring wells. In the upper 10-foot-depth interval, soil samples will be collected from 0 to 0.5, 4 to 6, and 8 to 10 feet bgs at all seven borings. At five of the borings, soil samples will also be collected from approximately 14 to 16 and 18 to 20 feet bgs. At the two monitoring well borings, up to 12 soil samples will be collected. From 10 to 30 feet bgs, soil samples will be collected at 5-foot sample intervals. And from 30 feet to groundwater, soil samples will be collected at 20-foot intervals.

### **4.2.2.3 Trenching**

Twelve trenches will be excavated using an all-terrain backhoe with extendable bucket boom at IRP Site 6, shown on Figure 2-1. The trenches will be 2 to 3 feet wide, 10 feet long, and will extend to an estimated depth of 10 feet bgs (contingent upon soil conditions to keep an open, stable trench). An average of three soil samples will be collected per trench, for up

to 36 samples. Sample collection depth will depend on site conditions; however, depths will include both surface (0 to 0.5 foot bgs) and subsurface sample locations. The locations of the soil samples will be biased to characterize the rubble debris. Soil samples will be collected from the excavator bucket using decontaminated or disposable scoops or trowels, as necessary.

Trench locations were chosen based on how well they allow mechanized equipment to access the sites requiring delineation of contaminants. Actual trench locations will be based in part on the results of the geophysical survey and visual observations of potential waste or debris. Prior to trenching, KCH field personnel will inspect the ground surface and overhead space to identify potential obstructions that could cause a safety hazard (e.g., subsurface utilities, overhead power lines, or trees). Site obstructions will be noted in the field so that KCH can set up the excavator at an alternate location, if needed, to mitigate potential safety hazards. Samples collected from trenches will be taken from the backhoe bucket; field personnel will not enter the trenches.

At each trench, the thickness of soil cover over the waste and soil classification will be documented. For any waste encountered, the character and content of the waste, amount of soil mixed with the waste, and the bottom depth of the waste will also be documented. Soil, waste, and any other significant features (e.g., burn ash) will be described and photographed, with observation details recorded in the logbook.

When sample collection is complete, the trench will be backfilled with the original material unless there are visible signs of contamination. Backfilled soil will be compacted with the backhoe bucket.

#### **4.2.2.4 Soil Sampling**

Soil samples will be collected directly from the hand auger, using a California modified split-spoon sampler lined with stainless steel sleeves, or from the excavator bucket. If samples require VOC or TPH-purgeable (TPH-p) analysis (based on visible contamination, odors, or PID readings in excess of 5 parts per million [ppm]), the soil sample will be collected from the end of the hand auger or stainless steel liner, at the appropriate depth, immediately after removal. Samples to be analyzed for VOCs and TPH-p will be collected using an EnCore (or equivalent) sampler for each sampling method. After the EnCore samples are collected, the remaining contents of the hand auger, the stainless steel liner, or the material from the excavator bucket will be emptied into a decontaminated, stainless steel mixing bowl. The soil in the bowl will be field-homogenized by mixing the soil with a decontaminated, stainless steel spoon or spatula. The remaining laboratory-supplied sample jars will be filled for submittal to the analytical laboratory.

Soil samples will be submitted to a United States Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP)-accredited analytical laboratory for the following analyses:

- SVOCs by United States Environmental Protection Agency (USEPA) 8270C.
- PAHs by USEPA 8270C selective ion monitoring (SIM).
- OCPs by USEPA 8081A.

- Polychlorinated biphenyls (PCBs) by USEPA 8082.
- Metals by USEPA 6020.
- Mercury by USEPA 7471A.
- Hexavalent chromium by USEPA 7199.
- VOCs by USEPA 8260B; contingency analysis analyzed at up to 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- TPH-p by USEPA 8015B; contingency analysis analyzed at up to 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- TPH-extractable (TPH-e) by USEPA 8015B; contingency analysis analyzed at up to 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- Dioxins/furans by USEPA 8290; contingency analysis analyzed at up to 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible evidence of burning (i.e., soot, ash, or other combustion byproducts).

A field decision to analyze more than 25 percent of samples for VOCs and TPH can be made if many samples have VOC and TPH indicators. Significant areas of VOC contamination without debris may be attributed to nearby USTs or previous spills.

Soil will be logged consistent with American Society for Testing and Materials International Method D 2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)*. A PID will be used to screen the soil for VOCs immediately upon retrieval, and the readings will be recorded on the soil boring logs.

Each hand auger borehole will be backfilled by pouring bentonite chips into the borehole to within a few inches of the surface and hydrating. Each HSA borehole will be backfilled with cement grout with 5 percent bentonite (pumped into the borehole from the bottom up with a tremie pipe) to within a few inches of the surface. All boreholes will be patched with native soil to match existing surface conditions.

### **4.2.3 Monitoring Well Installation**

Two monitoring wells will be installed at IRP Site 6 (see Figure 2-1) to evaluate whether groundwater beneath IRP Site 6 contains chemicals that pose an unacceptable risk to human receptors.

Well boreholes will be advanced using HSA drilling. Split-spoon samples will be collected every 5 feet with a California modified split-spoon sampler and will be logged by a California-registered geologist using the Unified Soil Classification System and recorded on logging forms. The first occurrence of groundwater (i.e. saturated sediments) will be recorded on the boring logs. One monitoring well will be installed into the uppermost saturated interval at each of the two proposed monitoring well locations.

Monitoring wells will be installed and constructed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981 and 1990). Each monitoring well will be constructed of 4-inch-diameter, Schedule 40, flush-jointed polyvinyl chloride (PVC) casing and factory-slotted well screen. Monitoring wells will be completed in a 10-inch-diameter borehole. Wells will be constructed within the first encountered groundwater. The total well depth is anticipated not to exceed approximately 150 feet bgs. A 30-foot-long well screen will be used (10 feet above and 20 feet below the water table) to accommodate changing regional water levels as a result of the current drought conditions. The well screen will have 0.020-inch slotted openings (four cuts per round). A monitoring well construction diagram is presented on Figure 4-1.

A filter pack of clean #3 Monterey sand will be poured from ground surface into the annular space between the auger flights and the well casing/well screen. If the well is constructed in an open borehole, then a tremie pipe will be used to emplace the filter pack. The filter pack will extend a minimum of 3 feet above the top of the screen. A 10-foot-thick layer (minimum) of hydrated bentonite (as chips or pellets) will be placed on top of the filter pack and allowed to fully hydrate before filling the remaining annular space with cement-bentonite grout.

A small notch will be cut on the northern side of the top of the PVC casing to be used as a reference for future water level measurements, and a water-tight PVC cap or equivalent will be placed on the top of the PVC casing. The well casing will be completed approximately 2 feet above ground surface. The aboveground well head completions will use an outer 5-foot-long protective steel surface casing, extending approximately 2.5 feet above ground surface with a locking cap, and surrounded by three or four protective steel bollards. A cement well pad approximately 3 feet square and a minimum of 4 inches thick that slopes away from the outer steel casing will be used. The well name will be clearly identified on the well pad or protective outer casing.

#### **4.2.4 Monitoring Well Development**

New monitoring wells will be developed by a drilling subcontractor prior to groundwater sampling. Monitoring well development will be performed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981 and 1990), using the surge-and-bail method followed by pumping as discussed in the following paragraph.

Groundwater monitoring wells will be developed by the surge-and-bail method and by pumping to improve hydraulic communication between the geologic formation and the well. Pumping will help remove suspended fines and result in monitoring wells that will produce sediment-free, low turbidity groundwater samples.

For each well, the development procedure will first include measuring the depth to groundwater and calculating the volume of water within the well casing. Subsequently, each of the wells will be surged using a surge block within approximately 5-foot intervals of the saturated portion of the screened interval, for approximately 10 minutes for each 5-foot interval. Next, the wells will be bailed or pumped to remove a minimum of three casing volumes of groundwater or until the pumped groundwater is free of visible turbidity and contains less than 10 nephelometric turbidity units (NTUs). Indicator parameters, including hydrogen ion concentration, temperature, turbidity, electrical conductivity, and dissolved

oxygen or oxidation-reduction potential will be monitored until they stabilize (Step 16 of Section 14.7) and/or extracted groundwater becomes visually clear and turbidity is less than 10 NTU, at which time development will be considered complete. The purge volumes, indicator parameters, and estimated recharge rates will be recorded in the field logbook during well development.

Development water will be transferred to United States Department of Transportation (DOT)-approved 55-gallon drums. The drums will be sealed, labeled, and stored in a secure location designated by the Navy.

#### **4.2.5 Groundwater Sampling**

The goal of groundwater sampling at IRP Site 6 is to evaluate whether COPCs have been adequately characterized in groundwater at IRP Site 6 and whether they pose an unacceptable risk to human receptors. New monitoring wells (MW-01 and MW-02) and existing monitoring wells (SB-10 and B-21), shown on Figure 2-1, will be monitored and sampled as indicated in the following discussion.

Groundwater monitoring will begin a minimum of 72 hours after completion of development. At each of the groundwater monitoring wells, groundwater samples will be collected consistent with the USEPA guidance *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (Puls and Barcelona, 1996). Details of the sampling procedure are provided in the SAP (Appendix A). Groundwater samples will be collected unfiltered (for total metals concentrations) and with the use of a 0.45-micron in-line disposable filter (for dissolved metals and hexavalent chromium concentrations). The preservative for filtered samples will not be added until after sample collection and filtration.

Groundwater samples will be submitted to a DoD ELAP-accredited analytical laboratory for the following analyses:

- SVOCs by USEPA 8270C
- PAHs by USEPA 8270C SIM
- OCPs by USEPA 8081A
- PCBs by USEPA 8082
- Total and dissolved metals by USEPA 6020
- Total and dissolved mercury by USEPA 7470A
- Hexavalent chromium by USEPA 218.6
- VOCs by USEPA 8260B
- TPH-p by USEPA 8015B
- TPH-e by USEPA 8015B

#### **4.2.6 Surveying**

All new trench, monitoring well, and borehole locations will be surveyed by a professional land surveyor, licensed by the State of California. The surveyor will provide the elevation at backfilled ground surface to a precision of 0.01 foot and its location to a precision of plus or minus 0.1 foot horizontally, based on the borehole center. The elevations will be surveyed relative to the 1988 National Geodetic Vertical Datum. The borehole and monitoring well locations will be surveyed using the 1983 North American Datum State Plane Coordinate System, California, Zone 5. Vertical coordinates will be reported as feet relative to msl.

#### **4.2.7 Investigation-Derived Waste**

IDW generated during this investigation will include soil cuttings from hand augering and HSA drilling, decontamination water, and purge water. All soil and water will be placed in DOT-approved 55-gallon drums or roll-off bins, sealed, labeled (drums), and stored in a secure location designated by the Navy until disposal can be secured at an offsite treatment or recycling facility. Sampling and analysis of IDW will be required for waste disposal profiling. Waste containment, labeling, sampling, and disposal will all be handled as part of IDW management. DFSP San Pedro will provide a secure waste storage facility. KCH will provide leak protection features (e.g., underlying leak proof floor or plastic sheet surrounded by a berm to contain leaks or rainfall runoff) for the temporary accumulation of IDW. Authorized DFSP San Pedro personnel will sign waste manifests. IDW is anticipated to be profiled as nonhazardous based on prior experience.

### **4.3 Safety and Health**

Field activities for this project will be performed in accordance with the SSHP and APP prepared for the proposed investigations.

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## 5.0 Reporting

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Results of the RI activities at IRP Site 6 will be summarized in an RI report, which will be reviewed and signed by a California-licensed professional geologist and other appropriate professionals. The report will include the following:

- Descriptions of field activities and methodologies used
- Summary of locations sampled
- Tabular summary of analytical results
- Summary of the extent of contamination (with supporting figures)
- Soil or hydrogeologic cross sections, if appropriate
- Soil boring and trench logs
- Photographs of field sampling and drilling locations and activities
- Well construction diagrams
- Groundwater elevation maps, if appropriate
- Site conceptual model
- Results of the HHRA and ERA
- Recommendations

Electronic data will be uploaded in Naval Electronic Data Deliverable format into the Naval Installation Restoration Information Solution.

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## 6.0 References

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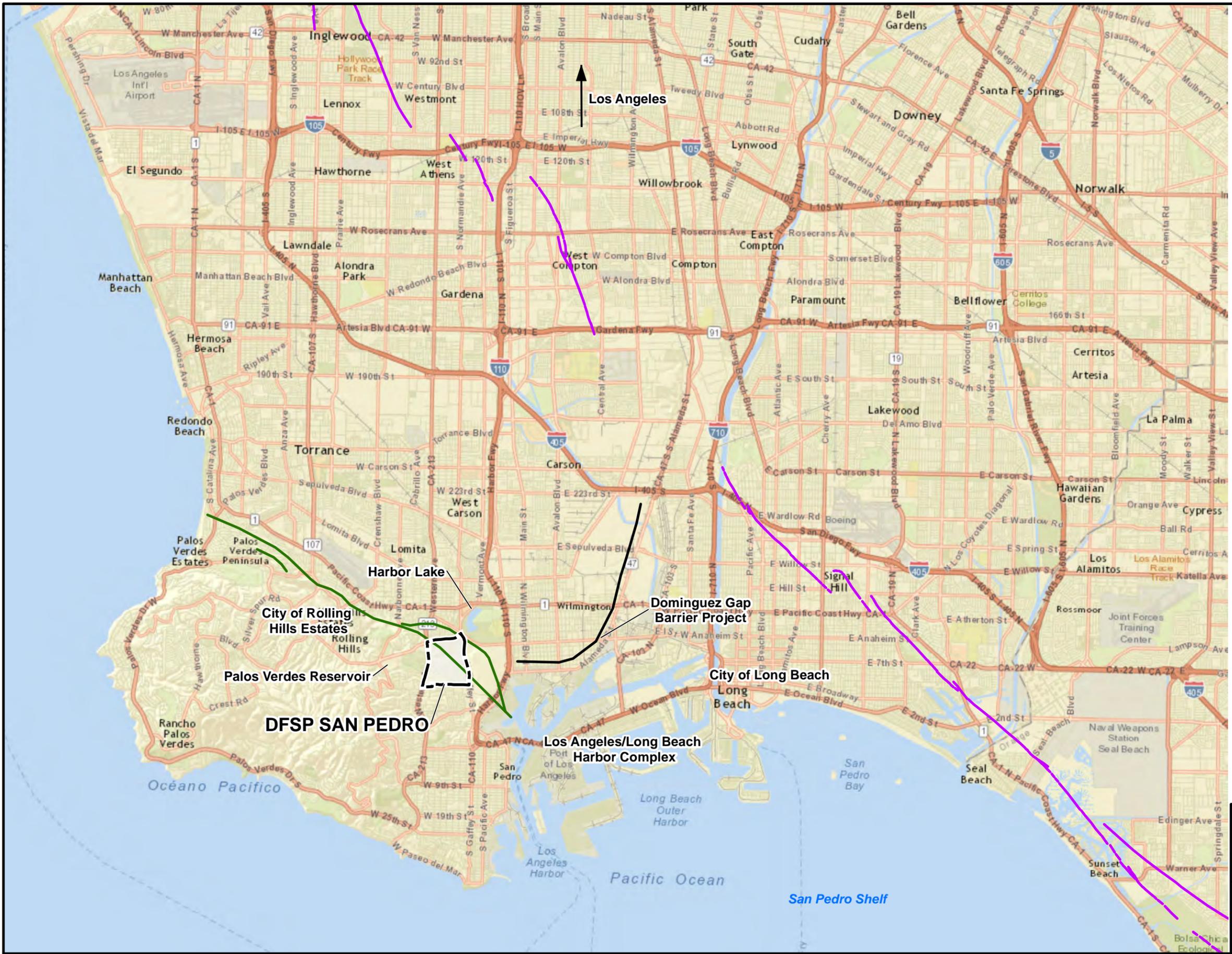
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## Figures

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- LEGEND**
-  NEWPORT-INGLEWOOD FAULT
  -  PALOS VERDES FAULT
  -  DFSP SAN PEDRO BOUNDARY

**NOTES:**  
 DFSP = Defense Fuel Support Point  
 IRP = Installation Restoration Program

**IMAGERY SOURCE:**  
 ESRI ArcGIS Online Web Service, Streets



**Site Vicinity**

Work Plan, Remedial Investigation at IRP Site 6  
Defense Fuel Support Point San Pedro, California



FIGURE  
**1-1**

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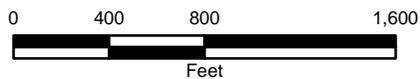
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**LEGEND**

-  IRP SITE BOUNDARY
-  FORMER NAVY HOUSING UNIT
-  DFSP SAN PEDRO BOUNDARY

NOTES:  
 - DFSP = Defense Fuel Support Point  
 - IRP = Installation Restoration Program



SOURCE:  
 ESRI ArcGIS Online Web Service,  
 World Imagery 5/5/2010

**IRP Site 6 Location**

Work Plan, Remedial Investigation at IRP Site 6  
 Defense Fuel Support Point San Pedro, California



FIGURE

**1-2**

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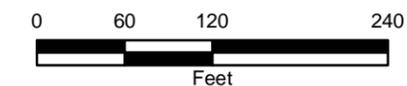
### LEGEND

- EXISTING GROUNDWATER MONITORING WELL
- HISTORICAL SOIL BORING
- PROPOSED HAND AUGER BORING
- PROPOSED SOIL BORING
- PROPOSED GROUNDWATER MONITORING WELL
- PROPOSED TRENCH
- IRP SITE 6 BOUNDARY

NOTE:  
- IRP = Installation Restoration Program

Approximate groundwater flow direction based on October 2013 groundwater elevation measurements (SGI, 2014).

IMAGERY SOURCE:  
ESRI ArcGIS Online Web Service, World Imagery  
5/25/2010



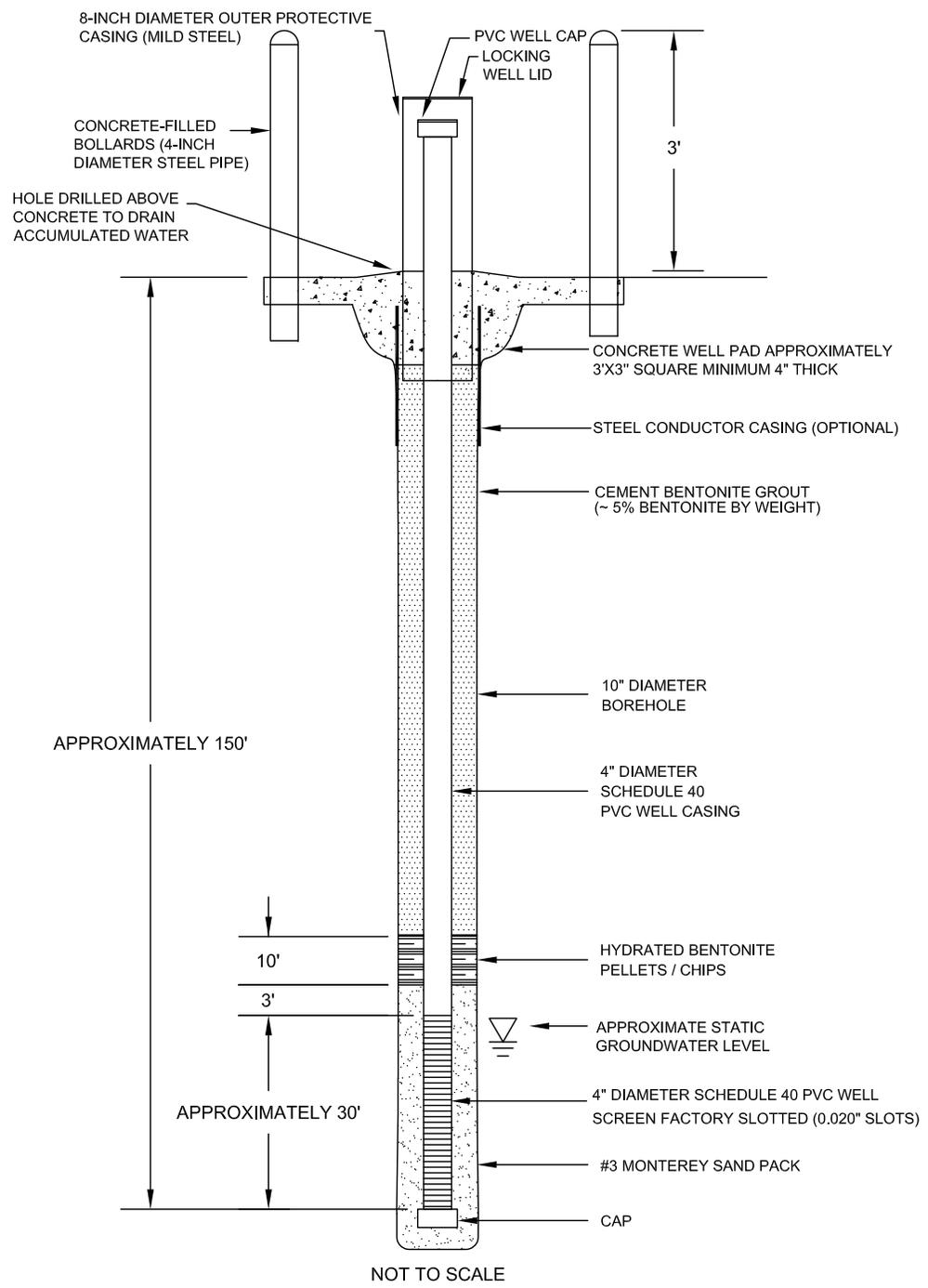
### IRP Site 6 Sample Locations

Work Plan, Remedial Investigation at IRP Site 6  
Defense Fuel Support Point San Pedro, California



FIGURE  
**2-1**

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NOTES:  
 PVC = Polyvinyl Chloride with threaded joints, no glued joints  
 IRP = Installation Restoration Program

**Proposed Groundwater Monitoring Well Construction Diagram**

Work Plan, Remedial Investigation at IRP 6  
 Defense Fuel Support San Pedro, California

 N		FIGURE <b>4-1</b>
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## **Appendix A**

# **Sampling and Analysis Plan**

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**SAP Worksheet #1—Title and Approval Page**

Draft

**Sampling and Analysis Plan  
(Field Sampling Plan and Quality Assurance Project Plan)  
Remedial Investigation at  
Installation Restoration Program Site 6  
Defense Fuel Support Point San Pedro, San Pedro, California**

June 2015

Prepared for



**Department of the Navy  
Naval Facilities Engineering Command  
Southwest**

Prepared by:



**CH2M HILL Kleinfelder, A Joint Venture  
1320 Columbia Street, Suite 310  
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Prepared under:

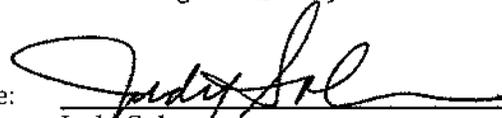
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Contract Task Order Number: CTO 0100  
Document Control Number: KCH-2622-0100-0009

Review Signature:

  
\_\_\_\_\_  
Theresa Rojas  
KCH Program Quality Assurance Manager

August 4, 2015  
Date

Approval Signature:

  
\_\_\_\_\_  
Judy Solomon  
NAVFAC LANT Chemist

8/5/15  
Date

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# Executive Summary

This document presents the Uniform Federal Policy (UFP) Tier I Sampling and Analysis Plan (SAP) for the Remedial Investigation (RI) to be performed at Installation Restoration Program (IRP) Site 6 at Defense Fuel Support Point (DFSP) San Pedro, located in Los Angeles, California. IRP Site 6 is a former disposal area referred to as the South Ravine. DFSP San Pedro's mission involves truck and pipeline transport of fuel to United States military units.

## Site History

During a 1990 site visit, wooden boards and furniture, brush, metal pipe, concrete, and tires were visible at IRP Site 6. Additionally, paint spills, rusted 55-gallon drums, and 1- and 5-gallon cans containing varying amounts of unidentified liquids were noted. Much of the debris was overgrown with vegetation.

The South Ravine has northwest and northeast branches. The northwest branch has been filled almost to grade. During the 1992 Site Inspection (SI), a fill depth of approximately 19 feet below ground surface (bgs) was identified in this branch. The northeast branch was described as incised (i.e., narrower with steeper sides), with visible fill (Jacobs, 1993).

The history of disposal in the South Ravine is unknown. DFSP San Pedro personnel were not aware of historical site activities that included the depositing of fill material, so disposal at IRP Site 6 may have been unscheduled (Jacobs, 1993).

## Previous Investigations

In 1981 or 1982, a fuel spill occurred at a nearby tank. An estimated 10,000 gallons of diesel fuel reportedly flowed east into the southern end of the South Ravine. No cleanup of the spill was attempted, but two soil borings were placed in the probable path of this spill. From these two borings, four soil samples were collected and analyzed for fuel hydrocarbons. No total fuel hydrocarbons were reported at concentrations above the detection limit of 10 milligrams per kilogram (Jacobs, 1993).

During the 1992 SI, miscellaneous debris, including metal, glass, and wood fragments, were present along the surface of the northeast branch of IRP Site 6. The upper portions of the northwest branch were covered with an asphalt-like substance, possibly the remnants of a fuel spill from Underground Storage Tank (UST) 5 that occurred in 1954 (Jacobs, 1993), which is to the northwest of IRP Site 6. Six borings were drilled at IRP Site 6 and analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), total petroleum hydrocarbons (TPH), organic lead, and metals. Constituents in soil were detected at concentrations above risk-based concentrations. The soil sample collected from boring B05 at 16.5 feet bgs contained the highest concentrations of VOCs, SVOCs, diesel, antimony, lead, and zinc. Groundwater was not sampled during the SI. The SI recommended that an RI/Feasibility Study (FS) be conducted at IRP Site 6.

## Planned Activities

During the RI at IRP Site 6, a geophysical survey will initially be conducted to assess the lateral extent of debris and to aid in the placement of sample locations. The survey will be focused in

the northern portion of IRP Site 6 where debris have been observed. Because of the presence of thick vegetation and steep terrain in the northeast branch, geophysical transect lines will be used rather than a traditional geophysical grid.

The sampling and analytical program for the RI at IRP Site 6 consists of 24 soil borings and trenches that will be installed throughout IRP Site 6 and sampled as follows:

- Five borings installed via hand auger to an estimated depth of 10 feet bgs, each with one surface soil sample collected between ground surface and 0.5 foot bgs and two subsurface soil samples (one each between 4 and 6 feet bgs and 8 and 10 feet bgs).
- Five borings installed by hollow-stem auger to an estimated depth of 20 feet bgs, each with one surface soil sample collected between ground surface and 0.5 foot bgs and four subsurface soil samples (one each from 4 to 6 feet bgs, 8 to 10 feet bgs, 14 to 16 feet bgs, and 18 to 20 feet bgs).
- Twelve 10-foot-long trenches will be excavated to an approximate depth of 10 feet bgs, with an average of three soil samples collected per trench. Sample collection depth will depend on site conditions; however, surface (0 to 0.5 foot bgs) and subsurface samples will be collected.
- Two borings will be advanced to an estimated depth of 150 feet bgs and monitoring wells will be installed in each boring. During well installation, up to 12 soil samples will be collected from each boring. Soil samples will be collected from 0 to 0.5 foot bgs, 4 to 6 feet bgs, and 8 to 10 feet bgs. Additional soil samples will be collected at 5-foot intervals from 10 feet to 30 feet bgs and at 20-foot intervals to groundwater. The monitoring wells will be developed prior to sample collection.
- Two new groundwater monitoring wells and two existing groundwater monitoring wells, one upgradient (SB-10) and one downgradient (B-21) from IRP Site 6, will be sampled during two events. Groundwater monitoring wells SB-10 and B-21 were installed in 1994 and 1995, respectively, as part of the assessment of the South Tank Farm (SGI, 2013).

Soil and groundwater samples will be analyzed as follows:

- Soil - SVOCs by United States Environmental Protection Agency (USEPA) 8270C, polycyclic aromatic hydrocarbons (PAHs) by USEPA 8270C selective ion monitoring (SIM), OCPs by USEPA 8081A, PCBs by USEPA 8082, metals by USEPA 6020A/7471A, and hexavalent chromium by USEPA 7199. Up to 25 percent (and a minimum of 10 percent) of the total samples will be analyzed for VOCs by USEPA 8260B, TPH-purgeable (TPH-p) by USEPA 8015B, and TPH-extractable (TPH-e) by USEPA 8015B. Samples will be selected for VOC, TPH-p, and TPH-e analyses if there is visible solvent contamination, odors, or photoionization detector readings in excess of 5 parts per million. Soils will also be analyzed for dioxins/furans by USEPA 8290A at a rate of up to 25 percent (and a minimum of 10 percent) of the total samples and will be selected if there is evidence of burn ash or incineration products.
- A field decision to analyze more than 25 percent of soil samples for VOCs, TPH-p, and TPH-e can be made if there is an elevated number of soil samples containing constituents

related to VOC, TPH-p, or TPH-e. Significant areas of VOC impact without debris may be attributed to nearby USTs or previous spills.

- Groundwater - VOCs by USEPA 8260B, SVOCs by USEPA 8270C, PAHs by USEPA 8270C SIM, TPH-p by USEPA 8015B, TPH-e by USEPA 8015B, OCPs by USEPA 8081A, PCBs by USEPA 8082, total and dissolved metals by USEPA 6020A/7470A, and hexavalent chromium by USEPA 218.6.

## Organization of the SAP

This SAP is organized according to the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) (USEPA, 2005). The UFP-QAPP is the outcome of the Intergovernmental Data Quality Task Force and is the format required by Naval Facilities Engineering Command Southwest under Environmental Work Instruction No. 2. The UFP-QAPP is the companion to the *Uniform Federal Policy for Implementing Environmental Quality Systems* (UFP-QS). The UFP-QS was developed to consistently implement the quality system requirements of American National Standards Institute (ANSI)/American Society for Quality E4-2004 Quality Systems for Environmental Data and Technology Programs (ANSI, 2004).

A list of the 37 UFP-QAPP worksheets included in this Tier I SAP is provided in the table of contents and Worksheet #2.

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## **Attachments**

- Attachment 1 Site Photos
- Attachment 2 Historical Soil Boring Locations and Analytical Data (Jacobs, 1993)
- Attachment 3 KCH SOPs
- Attachment 4 KCH Soil Boring Log
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- Attachment 6 Groundwater Sampling Data Sheet
- Attachment 7 Laboratory SOPs
- Attachment 8 Laboratory Certifications/ Accreditations

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# Acronyms and Abbreviations

amu	atomic mass unit
ANSI	American National Standards Institute
APP	Accident Prevention Plan
ASTM	American Society for Testing and Materials International
BAMP	biological avoidance and minimization plan
bgs	below ground surface
BHC	benzene hexachloride
°C	degree(s) Celsius
CA	corrective action
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCV	continuing calibration verification
CDFW	California Department of Fish and Wildlife
CDPH	California Department of Public Health
CLP	Contract Laboratory Program
CO	Contracting Officer
COC	chain-of-custody
COD	coefficient of determination
COPC	chemical of potential concern
CSM	conceptual site model
CTO	Contract Task Order
CVAA	cold vapor atomic absorption
%D	percent difference
DCB	decachloro biphenyl
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethene
DDT	dichlorodiphenyltrichloroethane
DFSP	Defense Fuel Support Point
DL	detection limit
DO	dissolved oxygen
DoD	United States Department of Defense
DOT	United States Department of Transportation
DQA	data quality assessment
DQI	data quality indicator
DQO	data quality objective
DTSC	California Department of Toxic Substances Control
EB	equipment blank
Eco-SSL	ecological soil screening level
EC	electrical conductivity
EDD	electronic data deliverable

EDL	estimated detection limit
EICP	extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
EMPC	estimated maximum potential concentration
ERA	ecological risk assessment
ESL	Environmental Screening Level
EWI	Environmental Work Instruction
FCR	field change request
FS	feasibility study
g	gram
GC/MS	gas chromatography/mass spectroscopy
GIS	geographic information system
HCl	hydrochloric acid
HHRA	human health risk assessment
HNO <sub>3</sub>	nitric acid
HpCDD	heptachlorodibenzo-p-dioxin
HpCDF	heptachlorodibenzofuran
HRGC	high resolution gas chromatography
HRMS	high resolution mass spectrometry
HSA	hollow-stem auger
HxCDD	hexachlorodibenzo-p-dioxin
HxCDF	hexachlorodibenzofuran
IC	ion chromatography
ICAL	initial calibration
ICB	initial calibration blank
ICP-MS	inductively-coupled plasma mass spectroscopy
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IDW	investigation-derived waste
IRP	Installation Restoration Program
IP/FP	implementation plan and fee proposal
IS	internal standard
KCH	CH2M HILL Kleinfelder, A Joint Venture
L	liter
L/min	liter per minute
LCS	laboratory control sample
LCSD	LCS duplicate
LOD	limit of detection
LOQ	limit of quantitation

µg/kg	microgram per kilogram
µg/L	microgram per liter
MB	method blank
MCL	maximum contaminant level
ME	marginal exceedance
mg/kg	milligram per kilogram
mg/L	milligram per liter
mL	milliliter
MS	matrix spike
MSD	matrix spike duplicate
msl	mean sea level
MW	monitoring well
N	normal
NA	not applicable
NAVFAC	Naval Facilities Engineering Command
Navy	United States Department of the Navy
NEDD	Naval Electronic Data Deliverable
NELAP	National Environmental Laboratory Accreditation Program
NIRIS	Naval Installation Restoration Information Solution
NTU	nephelometric turbidity units
OCDD	octachlorodibenzo-p-dioxin
OCDF	octachlorodibenzofuran
OCF	organochlorine pesticide
ORP	oxidation reduction potential
oz	ounce
PAH	polycyclic aromatic hydrocarbon
PAL	project action limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl
pdf	portable document format
PeCDD	pentachlorodibenzo-p-dioxin
PeCDF	pentachlorodibenzofuran
pH	hydrogen ion concentration
PID	photoionization detector
PM	Project Manager
POC	point of contact
ppm	parts per million
PQAO	Project Quality Assurance Officer
PSHM	Program Safety and Health Manager
PT	proficiency testing
PVC	polyvinyl chloride
QA	quality assurance

QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
QSM	Quality Systems Manual for Environmental Laboratories
%R	percent recovery
RF	radio frequency
RI	remedial investigation
RPD	relative percent difference
RPM	Remedial Project Manager
RSD	relative standard deviation
RSL	regional screening level
RT	retention time
RTC	response to comments
RWQCB	Regional Water Quality Control Board, Los Angeles Region
SAP	Sampling and Analysis Plan
SDG	sample delivery group
SFBRWQCB	San Francisco Bay Regional Water Quality Control Board
SI	Site Inspection
SIM	selective ion monitoring
S/N	signal to noise
SOP	standard operating procedure
SPCC	system performance check compound
SSHO	Site Safety and Health Officer
SSHIP	Site Safety and Health Plan
SVOC	semivolatile organic compound
TB	trip blank
TBD	to be determined
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzofuran
TOM	Task Order Manager
TPH	total petroleum hydrocarbons
TPH-e	total petroleum hydrocarbons-extractable
TPH-p	total petroleum hydrocarbons-purgeable
TSA	technical systems audit
UFP	Uniform Federal Policy
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
UFP-QS	Uniform Federal Policy for Implementing Environmental Quality
USA	Underground Service Alert
USEPA	United States Environmental Protection Agency
UST	underground storage tank
VOC	volatile organic compound

## SAP Worksheet #2—Sampling and Analysis Plan Identifying Information

**Site Name/Number:** Defense Fuel Support Point (DFSP) – Installation Restoration Program (IRP) Site 6

**Operable Unit:** Not applicable

**Contractor Name:** CH2M HILL Kleinfelder, A Joint Venture (KCH)

**Contract Number:** N62473-09-D-2622

**Contract Title:** Remedial Investigation (RI) at Installation Restoration Program (IRP) Site 6 Defense Fuel Support Point, San Pedro, California

**Work Assignment Number:** Contract Task Order (CTO) Number 0100

1. This sampling and analysis plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans* (USEPA, 2005), *USEPA Guidance for Quality Assurance Project Plans* (USEPA, 2002a), and Draft Final Naval Facilities Engineering Command Southwest (NAVFAC Southwest)-specific UFP-SAP Template (NAVFAC Southwest, 2011).

2. Regulatory program:  
 Comprehensive Environmental Response, Compensation, and Liability Act

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

The United States Department of the Navy (Navy) and KCH held a project kickoff meeting on May 28, 2014. Site visits were conducted with the Navy on April 9 and June 6, 2014.

5. List dates and titles of documents that are relevant to the current investigation:

Previous site work relevant to the current investigation is summarized in the following chart. Worksheet #10 includes a summary of the findings from previous investigations.

Reference Title	Date	Author
Final Site Inspection, Naval Fuel Support Point San Pedro, California	1993	Jacobs Engineering
Final Remedial Investigation Report for Installation Restoration Program Site 32, Southeast Ravine, Defense Fuel Support Point, San Pedro, California	2013	TriEco-Tt

6. Organizational partners (stakeholders) and connection with lead organization:

The stakeholders include the public (represented by the Restoration Advisory Board), the California Department of Toxic Substances Control (DTSC), Regional Water Quality Control Board, Los Angeles Region (RWQCB), and the California Department of Fish and Wildlife (CDFW). The DTSC, RWQCB, and CDFW oversee the cleanup process, which is being conducted by the Navy.

7. Lead organization:

The lead organization for the project is the Navy. The Navy uses the information gathered to make decisions in conjunction with the stakeholders.

8. If required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion in the following chart.

A list of the worksheets in this SAP is provided in the following chart.

**UFP-QAPP Crosswalk**

<b>UFP-QAPP Worksheet #</b>	<b>Required Information</b>	<b>Crosswalk to Related Information (if applicable)</b>
<b>A. Project Management</b>		
<i>Documentation</i>		
<b>1</b>	Title and Approval Page	
<b>2</b>	SAP Identifying Information	
<b>3</b>	Distribution List	
<b>4</b>	Project Personnel Sign-off Sheet	
<i>Project Organization</i>		
<b>5</b>	Project Organizational Chart	
<b>6</b>	Communication Pathways	
<b>7</b>	Personnel Responsibilities Table	
<b>8</b>	Special Personnel Training Requirements Table	
<i>Project Planning / Problem Definition</i>		
<b>9</b>	Project Scoping Session Participants Sheet	
<b>10</b>	Conceptual Site Model	
<b>11</b>	Project Quality Objectives/Systematic Planning Process Statements	
<b>12</b>	Field Quality Control Samples	
<b>13</b>	Secondary Data Criteria and Limitations Table	
<b>14</b>	Summary of Project Tasks	
<b>15</b>	Reference Limits and Evaluation Tables	
<b>16</b>	Project Schedule/Timeline Table	
<b>B. Measurement Data Acquisition</b>		
<i>Sampling Tasks</i>		
<b>17</b>	Sampling Design and Rationale	
<b>18</b>	Location-Specific Sampling Methods/SOP Requirements Table	
<b>19</b>	Field Sampling Requirements Table	
<b>20</b>	Field QC Sample Summary Table	
<b>21</b>	Project Sampling SOP References Table	See Worksheet #14
<b>22</b>	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	

**UFP-QAPP Crosswalk**

<b>UFP-QAPP Worksheet #</b>	<b>Required Information</b>	<b>Crosswalk to Related Information (if applicable)</b>
<i>Analytical Tasks</i>		
<b>23</b>	Analytical SOP References Table	
<b>24</b>	Analytical Instrument Calibration Table	
<b>25</b>	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	
<i>Sample Collection</i>		
<b>26</b>	Sample Handling System	
<b>27</b>	Sample Custody Requirements	
<i>Quality Control Samples</i>		
<b>28</b>	Laboratory QC Samples Table	
<i>Data Management Tasks</i>		
<b>29</b>	Project Documents and Records Table	
<b>30</b>	Analytical Services Table	
<b>C. Assessment Oversight</b>		
<b>31</b>	Planned Project Assessments Table	
<b>32</b>	Assessment Findings and Corrective Action Responses Table	
<b>33</b>	QA Management Reports	
<b>D. Data Review</b>		
<b>34 - 36</b>	Data Verification and Validation (Steps I and IIa/IIb) Process Table	
<b>37</b>	Usability Assessment	

Notes:  
 COC = chain-of-custody  
 QA = quality assurance  
 QC = quality control  
 SOP = standard operating procedure

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### SAP Worksheet #3—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Brenda Reese	Navy RPM	NAVFAC Southwest	(619) 532-4209	Brenda.Reese@navy.mil
Pei-Fen Tamashiro	Activity POC	NAVFAC Southwest	(562) 626-7897	pei-fen.tamashiro@navy.mil
Diane Silva	Command Records Manager	NAVFAC Southwest	(619) 532-3676	diane.silva@navy.mil
Alan Hsu	Lead RPM	DTSC	(714) 484-5395	ahsu@dtsc.ca.gov
Ann Lin	Lead RPM	RWQCB	(213) 576-6781	alin@waterboards.ca.gov
Eric Johansen	TOM	KCH	(619) 831-4605	ejohansen@kleinfelder.com
Theresa Rojas	Program QAM	KCH	(678) 530-4297	theresa.rojas@ch2m.com
Karin Kaiser	Project Chemist	KCH	(720) 612-0485	kkaiser@kleinfelder.com
Jerry Kellar	Data Manager	KCH	(619) 831-4537	gkellar@kleinfelder.com
Randy Kellerman	PQAO	KCH	(714) 435-6381	randy.kellerman@ch2m.com
Jeremiah Stock	Technical Lead/Field Manager	KCH	(619) 694-5514	JStock@kleinfelder.com
Bill Bergeron	SSHO	KCH	(781) 572-4574	bbergeron@kleinfelder.com
Ye Myint	Analytical Laboratory PM	EMAX	(310) 618-8889	ymyint@emaxlabs.com
Cynthia Clark	Analytical Laboratory PM	APPL	(559) 275-2175	cclark@applinc.com
Pei Geng	Data Validation PM	LDC	(760) 827-1100	pgeng@lab-data.com

Notes:

LDC = Laboratory Data Consultants  
 PM = Project Manager  
 POC = point of contact  
 PQAO = Project Quality Assurance Officer

RPM = Remedial Project Manager  
 SSHO = Site Safety and Health Officer  
 TOM = Task Order Manager

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### SAP Worksheet #4—Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Eric Johansen	KCH/TOM	(619) 831-4605			
Randy Kellerman	KCH/PQAO	(714) 435-6381			
Jeremiah Stock	KCH/Technical Lead/Field Manager	(619) 694-5514			
Karin Kaiser	KCH/Project Chemist	(720) 612-0485			
Jerry Kellar	KCH/Data Manager	(619) 831-4537			
Ye Myint	EMAX/Laboratory PM	(310) 618-8889			
Cynthia Clark	APPL/Laboratory PM	(559) 275-2175			
Pei Geng	LDC/Data Validation PM	(760) 827-1100			
Bill Bergeron	KCH/SSHO	(781) 572-4574			
TBD	KCH/Sampling Personnel	TBD			

**Notes:**

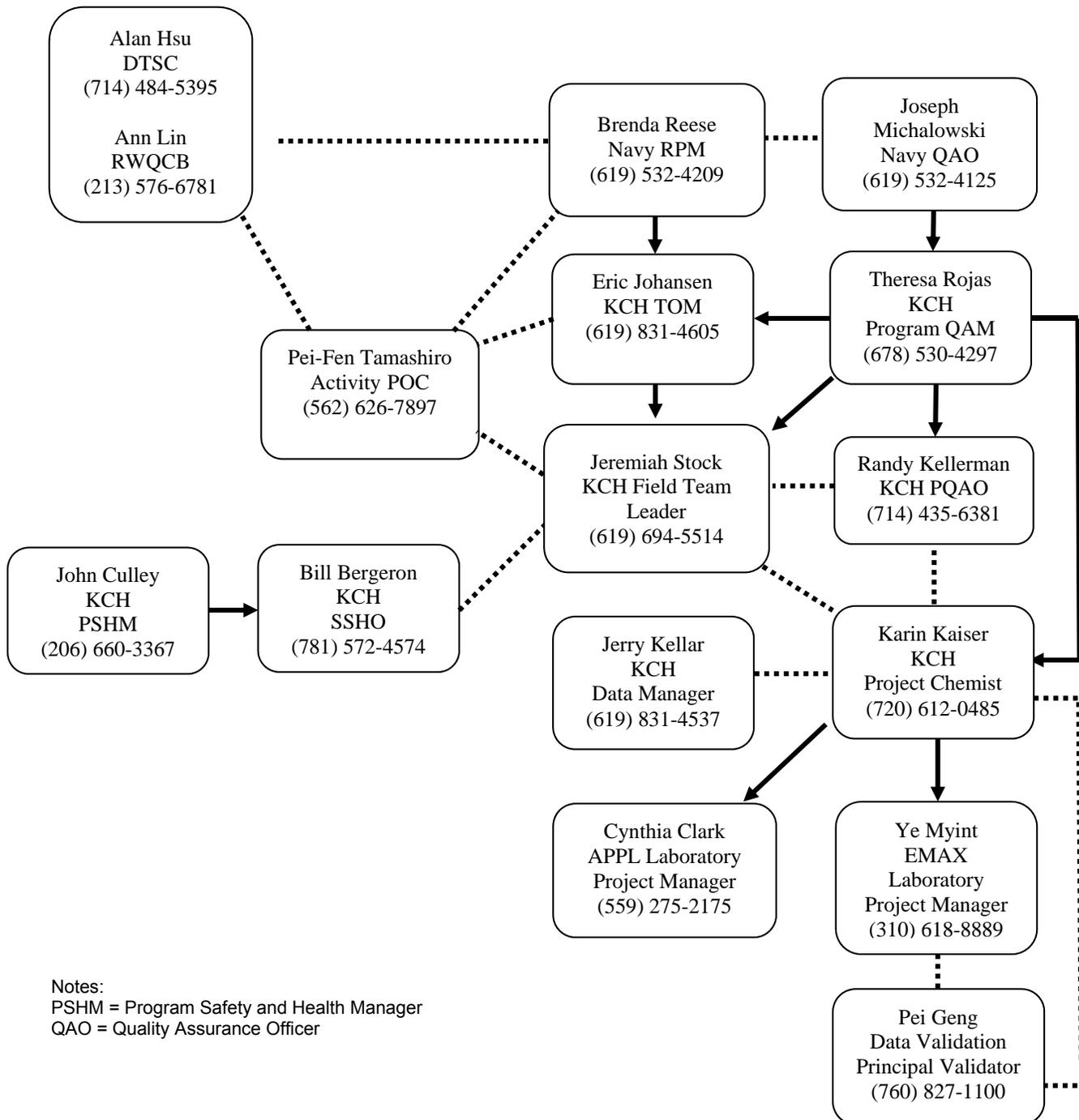
The sampling personnel will read the appropriate sections of this document before performing activities related to this SAP. The completed sign-off worksheet is maintained in the KCH project file.

TBD = to be determined

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## SAP Worksheet #5—Project Organizational Chart

Lines of Authority ————— Lines of Communication



Notes:  
 PSHM = Program Safety and Health Manager  
 QAO = Quality Assurance Officer

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## SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure (timing, pathway to and from, etc.)
Authorization for KCH to initiate field work	Navy RPM	Brenda Reese	(619) 532-4209	KCH TOM communicates verbally or by e-mail earliest schedule possible for fieldwork to commence. Navy RPM provides KCH TOM with written instruction to proceed upon completing coordination with Navy CO.
DFSP POC with Navy RPM, KCH TOM, and regulatory agencies	Activity POC	Pei-Fen Tamashiro	(562) 626-7897	Reports and other project-related information are forwarded to Activity POC by the TOM. Acts as a liaison between the Navy RPM, KCH TOM, other Navy departments, and regulatory agencies.
DTSC POC with Navy RPM	DTSC	Alan Hsu	(714) 484-5395	Reports and other project-related information are submitted by the Navy for review and comments by the agency.
RWQCB POC with Navy RPM	RWQCB	Ann Lin	(213) 576-6781	Reports and other project-related information are submitted by the Navy for review and comments by the agency.
KCH POC with Navy RPM	TOM KCH	Eric Johansen	(619) 831-4605	Materials and information about the project are forwarded to the Navy RPM by the TOM.
POC with Navy QAO	Program QAM KCH	Theresa Rojas	(678) 530-4297	Quality-related materials and information about the project are forwarded to the Navy QAO by the Program QAM.
SAP amendments	Program QAM KCH	Theresa Rojas	(678) 530-4297	Changes to the SAP are submitted in writing to the Navy QAO, who must approve the changes prior to implementation.
SAP amendment approvals	Navy QAO	Joseph Michalowski	(619) 532-4125	Issues final approval of SAP amendments to Program QAM via signed approval form (pdf is acceptable). Concurrence from the Navy RPM.
Revising sampling program (adding or removing sampling location or revising analytical suite)	TOM KCH	Eric Johansen	(619) 831-4605	Changes to the sampling program are submitted in writing as an FCR or proposed SAP amendment to the Navy QAO, who must approve the changes prior to implementation.

## SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure (timing, pathway to and from, etc.)
Field or analytical CAs	Program QAM KCH	Theresa Rojas	(678) 530-4297	The need for CAs is assessed by the Program QAM, who notifies the PQAQ and Navy QAO by phone or e-mail within 2 business days. PQAQ notifies the Navy RPM, TOM, and Field Manager (field issues) or Project Chemist (analytical issues) by phone or e-mail within 2 business days. The Navy RPM notifies DTSC by phone or e-mail within 2 business days of their notification.
Field progress reports	Field Manager KCH	Jeremiah Stock	(619) 694-5514	Daily field progress reports will be prepared by the Field Manager and submitted to the TOM, Navy RPM, and Navy Base POC by phone or e-mail.
Stop work issues	Field Manager KCH	Jeremiah Stock	(619) 694-5514	Field Manager notifies TOM about stopped work that occurs. All field personnel have stop work authority based on the APP and SSHP. Joseph Michalowski, Navy QAO, or representative, has authority to stop work if quality-related compliance issues are identified, or if there is noncompliance with field QC protocols, as specified in this SAP.
	Navy QAO	Joseph Michalowski	(619) 532-4125	
Field implementation of SAP changes	TOM KCH	Eric Johansen	(619) 831-4605	TOM notifies Field Manager by phone and e-mail of changes at least 2 days prior to field implementation.
Release of field data	Field Manager KCH	Jeremiah Stock	(619) 694-5514	Field data are reviewed by the Field Manager and are transmitted by e-mail or hard-copy shipping to the TOM.
Field deviations from the SAP	Field Manager KCH	Jeremiah Stock	(619) 694-5514	Field Manager notifies TOM and Program QAM by phone or e-mail within 2 days of the SAP deviation (nature of deviation and technical justification), Program QAM notifies Navy QAO immediately, and an FCR may be requested by Navy QAO for review and approval prior to implementation.

## SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure (timing, pathway to and from, etc.)
Analytical deviations from the SAP or reporting analytical data quality issues	PM Analytical Lab	Ye Myint Cynthia Clark	(310) 618-8889 (559) 275-2175	Laboratory subcontractor notifies Project Chemist and Program QAM within 24 hours by phone or e-mail and documents SAP deviations in the final analytical data report.
Analytical data validation issues	PM Data Validator	Pei Geng	(760) 827-1100	Analytical data validation subcontractor notifies Project Chemist within 2 business days and documents issues in the data validation report.
Notification of nonusable analytical data	Program QAM KCH Project Chemist KCH	Theresa Rojas Karin Kaiser	(678) 530-4297 (720) 612-0485	If significant problems are identified by the laboratory or the project team that affect the usability of the data (i.e., the data are rejected or the DQOs are not met), the Program QAM or Project Chemist will notify the Navy RPM and the Navy QAO within 24 hours or the next business day.
Release of analytical data to KCH	Project Chemist/ Data Manager KCH	Karin Kaiser Jerry Kellar	(720) 612-0485 (619) 831-4537	No analytical data can be released until validated analytical data are approved by the Project Chemist or Program QAM.
Report submittal to regulatory agencies	Navy RPM	Brenda Reese	(619) 532-4209	Navy RPM receives report from KCH and submits to DTSC. Navy RPM also provides copies as appropriate to other Navy contractors.
Response to regulatory comments	Navy RPM	Brenda Reese	(619) 532-4209	Navy RPM receives regulatory comments on submitted work plan and field activity report and coordinates responses with KCH, as necessary.

Notes:

APP = accident prevention plan  
 CA = Corrective Action  
 CO = Contracting Officer  
 DQO = data quality objective  
 FCR = field change request  
 pdf = portable document format  
 QAM = Quality Assurance Manager  
 SSHP = Site Safety and Health Plan

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## SAP Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Brenda Reese	Navy RPM	NAVFAC Southwest	<ul style="list-style-type: none"> <li>• Performs project management</li> <li>• Oversees the project cost and schedule</li> <li>• Provides overall direction for project</li> <li>• Provides authorization for work to be performed</li> <li>• Acts as liaison with regulatory agencies, including submittal of documents</li> <li>• Acts as liaison with other Navy departments</li> <li>• Oversees protocols for disposition of IDW</li> </ul>	
Joseph Michalowski	Navy QAO	NAVFAC Southwest	<ul style="list-style-type: none"> <li>• Provides governmental oversight of the project QA program</li> <li>• Provides quality related directives through Navy CO Representative</li> <li>• Acts as POC for matters concerning QA and the Navy's laboratory QA program</li> <li>• Coordinates training on matters pertaining to generation and maintenance of quality of data</li> <li>• Authorizes the suspension of project execution if QA requirements are not adequately followed</li> </ul>	
Pei-Fen Tamashiro	Activity POC	NAVFAC Southwest	<ul style="list-style-type: none"> <li>• Acts as liaison with regulatory agencies, including submittal of documents</li> <li>• Acts as liaison with other Navy departments</li> <li>• Oversees protocols for disposition of IDW, signs waste manifests, oversees base access issues</li> <li>• Reviews reports and other project-related information and provides comments</li> </ul>	
Alan Hsu	PM	DTSC	<ul style="list-style-type: none"> <li>• Reviews reports and other project-related information submitted by the Navy RPM and provides comments</li> </ul>	
Ann Lin	PM	RWQCB	<ul style="list-style-type: none"> <li>• Reviews reports and other project-related information submitted by the Navy RPM and provides comments</li> </ul>	
Regina Donohoe	PM	CDFW	<ul style="list-style-type: none"> <li>• Reviews reports and other project-related information submitted by the Navy RPM and provides comments</li> </ul>	
John Culley	PSHM	KCH	<ul style="list-style-type: none"> <li>• Oversees preparation of company safety programs and compliance</li> <li>• Reviews APP/SSHP</li> <li>• Acts as a liaison between TOM and SSO</li> </ul>	

### SAP Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Dana Sakamoto	Program Manager	KCH	<ul style="list-style-type: none"> <li>• Issues and authorizes appointment letters describing duties/responsibilities and delegating authority</li> <li>• Issues stand-down order when necessary</li> <li>• Monitors and controls project through audits and surveillance of activities</li> <li>• Interfaces directly with the Navy to maintain awareness in planning and scheduling</li> </ul>	
Eric Johansen	TOM	KCH	<ul style="list-style-type: none"> <li>• Issues stand-down order when necessary</li> <li>• Establishes an overall records management system</li> <li>• Implements the approved project specific plans</li> <li>• Evaluates project-specific procedures and plans</li> <li>• Evaluates the project schedule and budget</li> <li>• Ensures completion of the project scope and deliverables</li> </ul>	
Theresa Rojas	Program QAM	KCH	<ul style="list-style-type: none"> <li>• Serves as a POC for the Navy QAO</li> <li>• Reviews and approves QA plans and revisions</li> <li>• Periodically evaluates the effectiveness of the QA plans by conducting surveillances, audits, or management assessments</li> <li>• Assigns, directs, and supports the QA staff</li> <li>• Trains, qualifies, and evaluates personnel according to the QA plans</li> <li>• Reviews project specific SAPs as required</li> <li>• Directs QA audits</li> <li>• Evaluates and selects qualified subcontract analytical laboratories and analytical data validation companies</li> <li>• Reviews field deviations from the SAP and associated FCRs</li> </ul>	

## SAP Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Bill Bergeron	SSHO	KCH	<ul style="list-style-type: none"> <li>• Implements the SSHP; verifies that field personnel have required training and attend daily safety meetings</li> <li>• Is lead for identifying, communicating, and, as appropriate, addressing CAs for encountered hazards not initially addressed in the SSHP</li> <li>• Communicates and reports health and safety issues to the PSHM</li> </ul>	
Jeremiah Stock	Field Manager	KCH	<ul style="list-style-type: none"> <li>• Directs field operations</li> <li>• Documents field activities on daily field progress reports</li> <li>• Reviews field sampling data</li> <li>• Prepares field deviations from the SAP</li> </ul>	
Randy Kellerman	PQAO	KCH	<ul style="list-style-type: none"> <li>• Liaison between Program QAM and Field Manager to maintain proper implementation of field-related SAP requirements</li> <li>• Performs TSA of field activities or assigns qualified designee</li> <li>• Implements analytical data QC procedures</li> <li>• Evaluates whether project specifications have been met</li> <li>• Audits field performance as required</li> <li>• Provides technical support to Project Chemist and Data Manager</li> </ul>	
Karin Kaiser	Project Chemist	KCH	<ul style="list-style-type: none"> <li>• Participates in development of project-specific SAP</li> <li>• Implements contract requirements for analytical data collection</li> <li>• Implements analytical data QC procedures</li> <li>• Reviews analytical data reports</li> <li>• Coordinates analytical laboratory and data validation subcontractors and prepares the scopes of work for each</li> <li>• Reviews analytical data validation reports</li> <li>• Supports report preparation and assesses whether project specifications have been met</li> </ul>	
Jerry Kellar	Data Manager	KCH	<ul style="list-style-type: none"> <li>• Imports sample and analytical data into a database system</li> <li>• Provides sample and analytical data for technical report production</li> <li>• Transmits validated analytical data to the NIRIS in the NEDD format</li> </ul>	

### SAP Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Ye Myint Cynthia Clark	Analytical Laboratory PM	EMAX APPL	<ul style="list-style-type: none"> <li>• Oversees analytical laboratory analyses and data reporting</li> <li>• Communicates sample issues, quality outliers, or CAs</li> </ul>	
Pei Geng	Analytical Data Validation/ Principal Chemist	LDC	<ul style="list-style-type: none"> <li>• Oversees validation of analytical data, preparation of analytical data validation reports, and EDD preparation with validation qualifiers</li> <li>• Communicates laboratory reporting issues, quality outliers, or CAs</li> </ul>	

Notes:

- EDD = electronic data deliverable
- IDW = investigation-derived waste
- NEDD = Naval Electronic Data Deliverable
- NIRIS = Naval Installation Restoration Information Solution
- TSA = technical systems audit

## **SAP Worksheet #8—Special Personnel Training Requirements Table**

No special training is required.

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## SAP Worksheet #9a—Project Scoping Session Participants Sheet

<b>Project Name:</b>	Remedial Investigation at IRP Site 6	<b>Site Name:</b>	IRP Site 6
<b>Projected Date(s) of Sampling:</b>	October 2015	<b>Site Location:</b>	DFSP San Pedro, California
<b>Project Manager:</b>	Eric Johansen		
<b>Date of Session:</b>	April 9, 2014		
<b>Scoping Session Purpose:</b>	Site Visit and Scoping Session		

<b>Name</b>	<b>Title</b>	<b>Affiliation</b>	<b>Phone #</b>	<b>Email Address</b>	<b>Project Role</b>
Margaret Wallerstein	Principal Scientist	NAVFAC Southwest	(562) 626-7838	Margaret.Wallerstein.ctr@navy.mil	Activity POC
Eric Johansen	TOM	KCH	(619) 831-4605	e johansen@kleinfelder.com	TOM

### Comments/Decisions:

KCH TOM and Activity POC performed a site walk to familiarize themselves with IRP Site 6, develop a general plan for proposed RI activities, and to take photographs of IRP Site 6.

### Action Items:

The information collected during the site walk was used to generate the implementation plan and fee proposal (IP/FP) for potential task order X099.

### Consensus Decisions:

The KCH TOM and Activity POC agreed that an RI would be necessary to further assess site conditions.

## SAP Worksheet #9b—Project Scoping Session Participants Sheet

<b>Project Name:</b>	Remedial Investigation at IRP Site 6	<b>Site Name:</b>	IRP Site 6
<b>Projected Date(s) of Sampling:</b>	October 2015	<b>Site Location:</b>	DFSP San Pedro, California
<b>Project Manager:</b>	Eric Johansen		
<b>Date of Session:</b>	May 28, 2014		
<b>Scoping Session Purpose:</b>	Project Kickoff		

<b>Name</b>	<b>Title</b>	<b>Affiliation</b>	<b>Phone #</b>	<b>Email Address</b>	<b>Project Role</b>
Margaret Wallerstein	Principal Scientist	NAVFAC Southwest	(562) 626-7838	Margaret.Wallerstein.ctr@navy.mil	Activity POC
Brenda Reese	Navy RPM	Navy	(619) 532-4209	Brenda.reese@navy.mil	Navy RPM
Eric Johansen	TOM	KCH	(619) 831-4605	ejohansen@kleinfelder.com	TOM
Jeremiah Stock	Senior Geologist	KCH	(619) 694-5514	jstock@kleinfelder.com	Technical Lead
Krysten DeBroka	Project Geologist	KCH	(619) 831-4680	kdebroka@kleinfelder.com	Geologist

### Comments/Decisions:

KCH and Navy personnel met to discuss the contract award, scope of work for the RI, schedule for field activities, and access and limitations to fieldwork such as the presence of sensitive species. Meeting minutes were prepared and sent to the RPM via email on June 4, 2014.

### Action Items:

The following are action items identified during the meeting (the names of individuals or entities responsible for leading them are in parentheses):

- Meet onsite June 6, 2014 for a site walk (KCH and Navy). KCH will send an Outlook appointment. (Eric)
- Submit a request for geographic information system (GIS) files to Andrea Baratie at [andrea.baratie@navy.mil](mailto:andrea.baratie@navy.mil). (Eric via Margaret)

- Mark up the DFSP San Pedro base map with correct site names for IRP sites. Return map to KCH to make updates. (Navy)
- Use the name “South Ravine” instead of “Central Ravine” for all report documentation. (KCH)
- Obtain a hardcopy of the final Site Inspection Report (Jacobs, 1993). (Navy). Please note that KCH has located a pdf copy of the report on compact disc.
- Check files for copy of the Woodward-Clyde document (1990). (Navy)
- Combine work plan, SAP, and biological avoidance and minimization plan (BAMP) into one document. (KCH)
- Send example BAMP or biological monitoring template to KCH. (Margaret)
- Check DFSP San Pedro reports prepared by the Source Group Inc. (SGI) on GeoTracker for information regarding depth to water and groundwater flow direction in the vicinity of IRP Site 6. (KCH)
- Condense and re-submit the project schedule to the Navy to begin fieldwork in early 2015 (before blue butterfly flight season). (Eric)

**Consensus Decisions:**

KCH will obtain and review historical documents, prepare planning documents, set up GIS base files, and put historical data into the database.

KCH and the Navy will meet onsite June 6, 2014 for a site walk.

## SAP Worksheet #9c—Project Scoping Session Participants Sheet

<b>Project Name:</b>	Remedial Investigation at IRP Site 6	<b>Site Name:</b>	IRP Site 6
<b>Projected Date(s) of Sampling:</b>	October 2015	<b>Site Location:</b>	DFSP San Pedro, California
<b>Project Manager:</b>	Eric Johansen		
<b>Date of Session:</b>	June 6, 2014		
<b>Scoping Session Purpose:</b>	Site Visit and Scoping Session		

<b>Name</b>	<b>Title</b>	<b>Affiliation</b>	<b>Phone #</b>	<b>Email Address</b>	<b>Project Role</b>
Brenda Reese	RPM	Navy	(619) 532-4209	Brenda.reese@navy.mil	Navy RPM
Margaret Wallerstein	Principal Scientist	NAVFAC Southwest	(562) 626-7838	Margaret.Wallerstein.ctr@navy.mil	Activity POC
Eric Johansen	TOM	KCH	(619) 831-4605	ejohansen@kleinfelder.com	TOM
Jeremiah Stock	Senior Geologist	KCH	(619) 694-5514	jstock@kleinfelder.com	Technical Lead
Bill Bergeron	Project Scientist	KCH	(619) 694-5517	bbergeron@kleinfelder.com	SSHO

### Comments/Decisions:

KCH and Navy personnel performed a site walk to allow potential field personnel to visit IRP Site 6 and further refine the scope of work.

### Action Items:

KCH will use the information gathered on the site walk to identify sample locations, biologically sensitive areas, and potential site hazards, and to provide a more thorough description of site conditions in the planning documents.

### Consensus Decisions:

The Navy and KCH agreed that the number of sample locations proposed in the IP/FP should be adequate to address site conditions.

The Navy and KCH agreed that careful planning would be necessary to avoid impacts to sensitive species and their habitats when mobilizing heavy equipment to sample locations.

## SAP Worksheet #10— Conceptual Site Model

DFSP San Pedro is located in the community of San Pedro, in the city and county of Los Angeles, California, east of the city of Rolling Hills Estates and west of the city of Long Beach, and occupies approximately 331 acres (Figure 10-1). It is bordered on the north and south by residential and commercial property, on the east by the ConocoPhillips oil refinery, and on the west by Green Hills Memorial Park (Figure 10-2). DFSP San Pedro became operational in 1943 with the primary mission of storage and distribution of fuels to support military operations in California, Arizona, and Nevada. The majority of the underground storage tanks (USTs) and associated fuel pipelines were installed in 1944. Additional operational activities included storage of small arms ammunition after World War II, and construction of a small arms pistol range, housing areas, and baseball fields. The Navy operated DFSP San Pedro until 1980, when operational responsibility was transferred to the Defense Energy Support Center, a branch of the Defense Logistics Agency. DFSP San Pedro's current mission involves truck and pipeline transport of fuel to United States military units.

This investigation will focus on IRP Site 6 - South Ravine (Figure 10-2).

### 10.1 Site Description and History

After a 1990 site visit, IRP Site 6 was defined and identified as the South Ravine, a former disposal area. Paint spills, rusted 55-gallon drums, and 1- and 5-gallon cans containing varying amounts of unidentified liquids were noted. Wooden boards and furniture, brush, metal pipe, concrete, and tires were also visible during the site visit. Site photos taken during the 2014 site visit are included in Attachment 1.

The South Ravine has northwest and northeast branches (Figure 10-3). The northwest branch has been filled almost to grade. During the 1992 site inspection (SI), a fill depth of approximately 19 feet below ground surface (bgs) was identified in this branch. The northeast branch was identified as being incised (narrower and with steeper sides), with fill visibly present as well (Jacobs, 1993).

Engineering of the south-sloping ravine bottom in IRP Site 6 was apparent in 1943 topographic map (Jacobs 1993). A concrete v-drain was present during the 1990 site visit in the south end of the ravine bottom. Sands present in the ravine bottom may be from the upper part of the ravine where vertically cut banks are visible.

The history of disposal in the South Ravine is unknown. DFSP San Pedro personnel did not know of historical site activities that included the depositing of fill material, so it is likely that disposal at IRP Site 6 was unscheduled (Jacobs, 1993).

### 10.2 Previous Investigations

A fuel spill occurred in 1981 or 1982 at a nearby tank. An estimated 10,000 gallons of diesel fuel reportedly flowed east into the southern end of the South Ravine. No cleanup of the spill was attempted; however, two soil borings (WCB-10 and WCB-11) were placed in the probable path of the spill (Figure 10-3). Four soil samples were collected from approximately 2 to 15 feet bgs from boring WCB-10 and 2 to 4 feet bgs from boring WCB-11, and were analyzed for total petroleum hydrocarbons (TPH) as gasoline, kerosene, diesel, jet propellant-4, and unknown hydrocarbons. There were no TPH detections at concentrations above the detection limit (DL) of 10 milligrams per kilogram (mg/kg) for the four samples submitted (Jacobs, 1993).

An SI was conducted at IRP Site 6 in 1992. The IRP Site 6, South Ravine, consists of a north-south trending drainage that divides into northwest and northeast branches in the northern portion of the site (Figure 10-3). During the 1992 SI, miscellaneous debris, including metal, glass, and wood fragments, were present along the surface of the northwest branch. The upper portions of South Ravine were covered with an asphalt-like substance, possibly the remnants of a fuel spill from UST 5 (Jacobs, 1993).

During the 1992 SI, six soil borings (B01 through B06) were installed within IRP Site 6 (Figure 10-3). The locations of the historical soil borings and a summary of the analytical data collected during the SI are provided in Attachment 2. During the boring installations, it was noted that the northeast and northwest branches were filled with approximately 10 to 20 feet of assorted fill debris. Broken glass fragments were encountered at boring B01 in the northern end of the South Ravine. At boring B05, volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) were detected in soil. Although the soil was logged as fill material to approximately 14 feet bgs, the lower portions of this interval may be alluvium with abundant residual fuel products. The soils were overlaid with silty soil fill materials.

The South Ravine axis was also capped with a granular fill sand that was apparently emplaced during installation of a concrete v-drain. The v-drain is confined to the South Ravine axis (Figure 10-3). At the southern end of the ravine in IRP Site 6, rip-rap is present along the v-drain in the vicinity of boring B06. No fill soils were encountered in boring B06. Boring B07 was drilled east of the northern part of IRP Site 6, where clean sands of the San Pedro Formation were encountered (Jacobs, 1993).

Sampling conducted during the 1992 SI at IRP Site 6 indicated that elevated levels of chemicals of potential concern (COPCs) were present in soil, particularly in the northwest branch. COPCs identified in soil during the SI include fuels, organic lead, metals, SVOCs, and organochlorine pesticides (OCPs). Groundwater was not sampled during the SI. An RI/FS was recommended for IRP Site 6 based on these exceedances.

### **10.3 Geology and Hydrogeology**

The geology and hydrogeology information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013), which is approximately 800 feet east of IRP Site 6 and shown on Figure 10-2.

#### **Regional Geologic Setting**

DFSP San Pedro is near the northern end of the Peninsular Ranges geomorphic province. The Santa Ana, San Jacinto, and Laguna Mountains are included in this region. These mountains are predominantly composed of Cretaceous Period (65 to 145 million years old) igneous rocks and Tertiary Period (2.6 million to 65 million years old) marine sedimentary rocks (Jacobs, 1993). The Peninsular Range geomorphic province is characterized by northwest-trending mountain ranges and valleys bounded by right-lateral strike-slip faults. The primary geologic faults include the Palos Verdes, Newport-Inglewood, Whittier-Elsinore, and San Jacinto. DFSP San Pedro is at the southwestern tip of the Los Angeles basin. The Palos Verdes Hills, rising to an elevation of about 1,300 feet, are the most prominent feature in the area.

The Palos Verdes fault extends from the offshore area of the San Pedro Shelf into the Los Angeles harbor area and then along the northeast margin of the Palos Verdes Hills to the

Redondo Beach area (Figure 10-1). The fault plane is steeply dipping to the west offshore and exhibits right lateral strike-slip motion. Onshore, the fault has uplifted the Palos Verdes Hills and thrust them over the Torrance Plain along a steep fault plane dipping 65 degrees to 70 degrees to the southwest (Schell, 2008). The exact onshore location of the Palos Verdes fault is not known; however, the main trace of the fault zone has been mapped as passing through the northern portion of DFSP San Pedro (Bechtel, 1995). Folding of the local rocks has resulted in northwest-trending anticlines and synclines. The Gaffey anticline trends through DFSP San Pedro, with the axis about 500 feet north of IRP Site 6 (California Geological Survey, 2003). Pleistocene history of movement of the Palos Verdes fault system is recorded by the presence of 13 distinct wave-cut marine terraces along the coastal front (Woodring et al., 1946). The marine terraces are wave-cut surfaces incised and successively uplifted to their present elevations. DFSP San Pedro is on Marine Terrace No. 1, the lowest-altitude terrace in the sequence of 13 wave-cut marine terraces.

Elevations across DFSP San Pedro vary from approximately 25 feet above mean sea level (msl) on the eastern side, to approximately 260 feet above msl in the west (SGI, 2009). The hills are cut by several ravines and gullies. The primary ravine (usually referred to as the Central Ravine), which is north of IRP Site 6, bisects DFSP San Pedro and trends east-west. DFSP San Pedro is underlain by metamorphic basement of late Jurassic- to late Cretaceous-age and sedimentary deposits ranging from Miocene to Holocene. The stratigraphy is characterized from youngest to oldest as follows (Bechtel, 1995):

- **Fill.** Manmade fill deposits are present throughout DFSP San Pedro. Most of these deposits are uncontrolled fill materials placed in former ravines, such as IRP Site 6. These materials consist primarily of loose to dense silty sand and clayey sand mixed with debris and construction rubble.
- **Alluvium.** Relatively thin deposits of Holocene alluvium are present in existing ravines and at depths along previously buried ravines. These soils are generally loose- to medium-dense, dark brown sand and silty sand.
- **Terrace Deposits (Lakewood Formation).** Late Pleistocene marine and nonmarine terrace deposits are locally found in DFSP San Pedro. These undifferentiated deposits can be up to 700 feet thick. The lower portion of the terrace deposits has been reported to consist of fairly continuous dense to very dense sand and gravel with finer-grained lenses, while the upper portion of the terrace deposits consists of predominantly fine-grained flood plain deposits with discontinuous lenses of variable lithologies (Wilkinson, 2003). These deposits have been grouped together and mapped in the area as the Lakewood Formation (Wilkinson, 2003; OHM Remediation Services Corp., 2001).
- **San Pedro Formation.** The Pleistocene (approximately 11,000 to 1.6 million years old) San Pedro Formation unconformably overlies the Monterey Formation. The San Pedro Formation generally consists of marine gravels, silty sands, and clays in stratified layers and some soft limestone. Bivalve shells are locally present in San Pedro Formation sediments. The San Pedro Formation appears at the ground surface across DFSP San Pedro.
- **Monterey Formation.** The Monterey Formation unconformably overlies the metamorphic basement. The Monterey Formation is middle to upper Miocene (5 million to about 15 million years old), composed of three sedimentary members of marine origin: Altamira

Shale, Valmonte Diatomite, and Malaga Mudstone. Malaga Mudstone is the uppermost member and is the only unit that has been encountered at DFSP San Pedro. This unit is exposed along the sidewalls of the upper Central Ravine and has been encountered at depth at DFSP San Pedro. Malaga Mudstone has been characterized in soil boring logs as a clayey siltstone with some thin sandstone interbeds and appears olive-green and weathered (Jacobs, 1993).

- **Metamorphic Basement.** Late Jurassic- to late Cretaceous-age (approximately 65 to 135 million years old) metamorphic rocks of the Franciscan Formation (Catalina schist facies) are typically hard, layered, and composed primarily of schist. The Franciscan Formation generally lies 1,200 to 4,000 feet beneath the Palos Verdes Hills.

## Regional Hydrology and Hydrogeology

The regional surface drainage flows west to east from the Palos Verdes Hills, across DFSP San Pedro, and into Los Angeles Harbor. Local surface water runoff is generally controlled by drainage through existing ravines, overland flow, and engineered drainage devices (Bechtel, 1995).

Three bodies of surface water are located near DFSP San Pedro: Harbor Lake, Palos Verdes Reservoir, and Los Angeles Harbor. Harbor Lake, formerly known as Bixby Slough, is located 0.25 mile northeast of DFSP San Pedro. Harbor Lake is a protected wildlife refuge and is part of the basin flood control system. The Palos Verdes Reservoir is located approximately 0.5 mile west of DFSP San Pedro. The reservoir is a covered treated-water storage facility for water supply. The California Water Service Company provides water from the reservoir to the Palos Verdes area, and the Los Angeles Department of Water and Power withdraws water from the reservoir to service parts of San Pedro. Los Angeles Harbor, located approximately 0.9 mile east of IRP Site 6, is bordered by heavy industrial facilities. The land surface in this area has been altered to facilitate industrial development. Los Angeles Harbor is used as a main corridor for military, commercial, and privately owned ocean vessels and commercial fishing operations.

Saltwater intrusion barriers are maintained along the Los Angeles and Orange County sections of the coastal plain. In Los Angeles County, imported and recycled water is injected to maintain a saltwater intrusion barrier (DWR, 2003). The closest of these saltwater intrusion barriers, known as the Dominguez Gap Barrier Project, is east of DFSP San Pedro. The Dominguez Gap Barrier Project began operation in 1971, and comprises a line of injection and observation wells extending 12 miles from F Street to E Street along the Dominguez Channel. The observation wells, used to monitor water surface elevations and depth-specific chloride levels, are located along the alignment of the barrier and are placed between injection wells or situated off of the immediate barrier alignment.

California DWR identified the three major aquifers in the West Coast Basin: the Silverado and Lynwood aquifers, part of the San Pedro Formation, and the Gage aquifer, part of the Holocene and latest Pleistocene deposits (DWR, 1961; Wilkinson, 2003). The Gage aquifer is the lowermost aquifer of the Lakewood Formation and is of upper Pleistocene age. The Lynwood and Silverado aquifers are lower Pleistocene age, and are the uppermost and lowermost aquifers in the San Pedro Formation, respectively. The San Pedro Formation contains the most extensive aquifers in the West Coast Basin and is the principal source of groundwater in the basin (Jacobs, 1993). The marine and continental deposits of the Lakewood Formation include the Gage aquifer, consisting of fine sand with interbedded silty and sandy clay, and silty and

clayey fine- to medium-grained sand. The Gage aquifer has been reported to be approximately 100 feet thick near DFSP San Pedro (OHM Remedial Services Corp., 2001). The hydrogeologic units used by DWR (DWR, 1961) that are well defined in the interior of the basin are less clear in the vicinity of DFSP San Pedro and the neighboring ConocoPhillips refinery (Wilkinson, 2003). For the purposes of the hydrogeologic evaluation and discussion, five hydrostratigraphic units have been defined for use at the neighboring ConocoPhillips Refinery (Wilkinson, 2003). The upper three units correlate to the Lakewood and upper San Pedro Formations and are believed to be representative of the Gage aquifer.

Depth to first-encountered groundwater is variable throughout DFSP San Pedro, ranging from approximately 10 to 35 feet bgs near the Pump House Area, to approximately 15 to 134 feet bgs at the Tank Farm Area, and 43 to 74 feet bgs in the Administrative Area (SGI, 2008).

The RWQCB adopted a basin plan (RWQCB, 1994) that designates groundwater beneath DFSP San Pedro for municipal and domestic beneficial use. Groundwater at DFSP San Pedro is not currently used for municipal or industrial purposes (no water production wells are present at DFSP San Pedro). The database of the Water Replenishment District (WRD) of Southern California indicates the presence of seven active supply wells within an approximately 4-mile radius of IRP Site 6 (WRD, 2009). The closest supply well is approximately 1 mile away at the ConocoPhillips refinery and is screened in the Silverado aquifer (Trihydro, 2008).

## 10.4 Climate

The climate information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013).

The climate in the southern California coastal region is semiarid and moderate. Seasonal variations are mild. Low clouds are predominant in the region in the early morning and at night. The most dramatic weather occurs during the winter, when patterns of rainstorms and Santa Ana winds (hot, dry winds that blow from the east) are possible. Based on climate records for the region, temperatures range from an average low of 44 degrees Fahrenheit (°F) in January to an average high of 79 °F in August. Average monthly precipitation ranges from 0.02 inch in July to 3.2 inches in February, with an average annual precipitation of 13.5 inches (Western Regional Climate Center, 2010).

## 10.5 Ecological Setting

The ecological setting information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013).

Vegetation at IRP Site 6 is anticipated to be similar to vegetation at IRP Site 32, which consists of chaparral and grassland species, including mustard (*Hirschfeldia incana*), thistle (*Centaurea melitensis*), and various wild grasses such as wild oat (*Avena fatua*). Previous reports included inventories of plants and animals found in and around DFSP San Pedro (Jacobs, 1993; OHM Remedial Services Corp., 2001), but surveys specific to IRP Site 6 were not performed.

Potential bird species at IRP Site 6 include the common raven (*Corvus corax*), American kestrel (*Falco sparverius*), American robin (*Turdus migratorius*), California towhee (*Pipilo crissalis*), European starling (*Sturnus vulgaris*), house finch (*Carpodacus mexicanus*), mourning dove (*Zenaidura macroura*), rock dove (*Columba livia*), and western meadowlark (*Sturnella neglecta*).

Mammals potentially present at IRP Site 6 include species of small rodents and larger mammals. Rodents include the California ground squirrel (*Spermophilus beecheyi*), deer mouse (*Peromyscus maniculatis*), dusky-footed woodrat (*Neotoma fuscipes*), fox squirrel (*Sciurus niger*), and western harvest mouse (*Reithrodontomys megalotis*). Larger mammals include the badger (*Taxidea taxus*), black-tailed jackrabbit (*Lepus californicus*), coyote (*Canis latrans*), gray fox (*Urocyon cinereoargenteus*), raccoon (*Procyon lotor*), red fox (*Vulpes vulpes*), and striped skunk (*Mephitis mephitis*).

A number of special status species could occur at IRP Site 6, including the federally listed threatened Coastal California gnatcatcher (*Polioptila californica californica*) and the federally listed endangered Palos Verdes Blue Butterfly (PVB) (*Glaucopsyche lygdamus palosverdensis*). The PVB was believed to be extinct for 11 years until discovered at DFSP San Pedro on March 10, 1994 (Mattoni, 1994). The PVB is closely associated with its larval food plants, California locoweed (*Astragalus trichopodes* var. *lonchus*) and common deerweed (*Lotus scoparius*). The PVB completes its entire life cycle near its host plant. Deerweed was observed at IRP Site 6 during a June 6, 2014 site walk.

## 10.6 Exposure Pathways

Following receipt of validated analytical data for the RI report, the conceptual site model (CSM) and potential exposure pathways will be refined for IRP Site 6, and screening-level human health and ecological risk assessments will be performed. If risk estimates for human and ecological receptors under current and unrestricted land use assumptions (human health evaluation only) are found to be acceptable, no land use controls or further action will be deemed necessary. A human health risk assessment (HHRA) will be completed using the RI data to evaluate potential risks from soil and groundwater exposure pathways. The HHRA will evaluate potential risks to commercial/industrial/military workers, construction workers, and hypothetical future residents from exposure to soil and groundwater from incidental ingestion, dermal contact, and inhalation of dust and vapors.

An ecological risk assessment (ERA) will also be completed using the RI data. The ERA will evaluate potential risk from exposure to soil. Analytical data from the RI will be compared with ecotoxicity benchmarks (literature-based) using conservative assumptions. A risk refinement step (Step 3a of ERA) also may be conducted if the ERA indicates unacceptable or uncertain risk. Step 3a is a re-evaluation of the conservative exposure assumptions of the ERA and assesses ecological risk based on more realistic assumptions such as average concentrations and life history parameters.

### SAP Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements

Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7
State the Problem	Identify the Goals of the Study	Identify Information Inputs	Define the Study Boundaries	Develop the Analytic Approach	Specify the Performance or Acceptance Criteria	Develop the Plan for Obtaining Data
<p>IRP Site 6 – the South Ravine – was a former disposal area containing wood, furniture, brush, metal pipe, concrete, and tires. Paint spills, rusted 55-gallon drums, and 1- and 5-gallon cans containing varying amounts of liquids were noted during a 1990 site visit.</p> <p>An SI was conducted at IRP Site 6 in 1992. Soil samples that were collected contained COPC concentrations that exceeded risk-based concentrations defined in the SI (Jacobs, 1993). Groundwater was not sampled during the SI. The SI recommended that an RI/FS be conducted at IRP Site 6.</p>	<ul style="list-style-type: none"> <li>Is the nature and extent of debris at IRP Site 6 adequately characterized?</li> <li>Is IRP Site 6 adequately delineated?</li> <li>Do concentrations of chemicals in soil or groundwater pose an unacceptable risk to human health or the environment?</li> <li>Is there evidence of VOCs, TPH, or dioxins/furans in soil in the trenches?</li> </ul>	<ul style="list-style-type: none"> <li>Site history, as described in the 1992 SI (Jacobs, 1993)</li> <li>Soil and groundwater final validated analytical data collected during this investigation</li> <li>Background analytical data (TriEco-Tt, 2013)</li> <li>Field observations, including visible contamination, odors, PID readings in excess of 5 ppm, and evidence of burning (combustion products) in soil samples</li> <li>HHRA inputs, including exposure pathways and assumptions for potential human receptors (current and future industrial and construction workers, hypothetical future residents)</li> <li>ERA inputs, including exposure pathways and assumptions for potential ecological receptors (plants, invertebrates, birds, and mammals)</li> </ul>	<p>The specific samples to be collected define the physical boundaries of the investigation and are set forth in Worksheet #18 and Figure 10-3.</p> <p>Soil samples will be collected to a maximum depth of approximately 120 feet bgs.</p> <p>Groundwater samples will be collected from depths ranging from approximately 120 feet bgs to 150 feet bgs.</p> <p>The temporal boundary for the IRP Site 6 investigation will be the time for completion of the Final RI, which is anticipated to be 2016. Temporal boundaries are subject to seasonal variations. Sampling activities need to avoid the breeding season of the PVB (approximately February 15 through May 30).</p>	<ul style="list-style-type: none"> <li>If the vertical and horizontal extent of debris layer is observed in the trenches, then the extent of debris is adequately characterized.</li> <li>If the vertical and horizontal extent of debris layer is not observed in the trenches, then the need for further investigation may be evaluated.</li> <li>If the horizontal and vertical extent of soil and groundwater contamination at IRP Site 6 is adequately delineated, then no additional samples will be collected.</li> <li>If the horizontal and vertical extent of soil and groundwater contamination at IRP Site 6 is not adequately delineated, then the need for further investigation will be evaluated.</li> <li>If the HHRA and ERA identify chemicals in soil or groundwater at concentrations that pose an unacceptable risk to human health or the environment, then additional evaluation may be recommended.</li> <li>If the HHRA and ERA do not identify chemicals in soil and groundwater that pose an unacceptable risk to human health or the environment, then a “no further action” designation will be considered.</li> <li>If there is visible contamination, odor, or PID readings in excess of 5 ppm, then soil samples will be analyzed for VOCs, TPH-p, and TPH-e. If there is visible evidence of burning, soil samples will be analyzed for dioxins/furans.</li> <li>If there is no visible contamination, odor, or PID readings in excess of 5 ppm, then soil samples will not be analyzed for VOCs, TPH-p, and TPH-e. If there is no visible evidence of burning, soil samples will not be analyzed for dioxins/furans.</li> </ul>	<p>Soil and groundwater sampling will be conducted using field sampling and processing methods, as described in Worksheet #14, and will be judgmentally located based on historical information. For reproducibility and comparability of analytical data, standard USEPA-approved analytical methods will be used. Samples will be analyzed by laboratories that are accredited by the DoD ELAP, NELAP, and CDPH ELAP.</p> <p>Performance criteria for the analytical methods to be performed for this investigation are established based on the DQIs, which include PARCCS. Sensitivity requirements are established by the PALs presented in Worksheet #15. Requirements for other DQIs are presented in Worksheets #12, #28, and #37.</p>	<p>The sampling design and rationale are discussed in detail in Worksheet #17</p> <p>Soil samples will be collected from 12 borings and 12 trenches at IRP Site 6. Soil samples will be collected from 0 to 0.5, 4 to 6, and 8 to 10 feet bgs in support of the HHRA and ERA at all borings, and deeper depth intervals to groundwater for characterization for the following analyses: SVOCs by USEPA 8270C, PAHs by USEPA 8270SIM, OCPs by USEPA 8081A, PCBs by USEPA 8082, metals by USEPA 6020A/7471A, and hexavalent chromium by USEPA 7199.</p> <p>VOCs by USEPA 8260B, TPH-p and TPH-e by USEPA 8015B, and dioxins/furans by USEPA 8290A are contingency analyses, analyzed at a frequency up to 25 percent (and a minimum of 10 percent) of the soil sample locations if there is visible contamination, odors, or PID readings in excess of 5 ppm, or visible evidence of burning (dioxins/furans).</p> <p>Groundwater samples will be collected from four monitoring wells during two sampling events. Groundwater samples will be analyzed for VOCs by USEPA 8260B, SVOCs by USEPA 8270C, PAHs by USEPA 8270SIM, TPH-p and TPH-e by USEPA 8015B, OCPs by USEPA 8081A, PCBs by USEPA 8082, total and dissolved metals by USEPA 6020A/7470A, and hexavalent chromium by USEPA 218.6.</p> <p>Quality control samples will be collected at required frequencies, as detailed in Worksheets #12 and #20.</p> <p>Analytical data will be validated by an independent, third-party contractor.</p>

Notes:

CDPH = California Department of Public Health  
 DoD = United States Department of Defense  
 DQI = data quality indicator  
 ELAP = Environmental Laboratory Accreditation Program  
 NELAP = National Environmental Laboratory Accreditation Program  
 PAH = polycyclic aromatic hydrocarbon  
 PAL = project action limit  
 PARCCS = precision, accuracy, representativeness, completeness, comparability, and sensitivity  
 PCB = polychlorinated biphenyl  
 PID = photoionization detector  
 SIM = selective ion monitoring  
 TPH-e = total petroleum hydrocarbons-extractable  
 TPH-p = total petroleum hydrocarbons-purgeable  
 USEPA = United States Environmental Protection Agency

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## SAP Worksheet #12a—Field Quality Control Samples – Groundwater

QC Sample	Analytical Group	Frequency	Data Quality Indicator	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A), or Both (S&A)
Field Duplicate <sup>a</sup>	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Dissolved Metals, Hexavalent Chromium	One per 10 field samples	Precision	RPD of less than 35 percent	S & A
Equipment Blank <sup>a</sup>	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Hexavalent Chromium	One per day of field sampling	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants (as defined in USEPA, 2014a)	S & A
Source Blank	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Hexavalent Chromium	One per water source	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants	S & A
Trip Blank	VOCs, TPH-p	One per cooler containing samples for VOC or TPH-p analysis	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants	S & A
Temperature Blank <sup>b</sup>	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Dissolved Metals, Hexavalent Chromium	One per cooler	Representativeness	≤ 6 °C, not frozen	S

Notes:

<sup>a</sup> Quality control sample analyses will be specific to the samples they are associated with.

<sup>b</sup> The laboratory will measure the temperature of the temperature blank upon receipt of the cooler. If the temperature is outside the acceptable range, the effect on the data quality of the sample results will be evaluated during data validation. No other chemical analyses will be performed on the temperature blank.

°C = degrees Celsius

LOQ = limit of quantitation

RPD = relative percent difference

## SAP Worksheet #12b—Field Quality Control Samples – Soil

QC Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A), or Both (S&A)
Equipment Blank <sup>a</sup>	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Hexavalent Chromium, Dioxins/Furans	One per day of field sampling	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants (as defined in USEPA, 2014a)	S & A
Source Blank	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Hexavalent Chromium, Dioxins/Furans	One per source	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants	S & A
Trip Blank	VOCs, TPH-p	One per cooler containing samples for VOC or TPH-p analysis	Accuracy	Target analytes < ½ the LOQ, with the exception of common laboratory contaminants	S & A
Temperature Blank <sup>b</sup>	VOCs, SVOCs, PAHs, TPH-p, TPH-e, OCPs, PCBs, Total Metals, Hexavalent Chromium, Dioxins/Furans	One per cooler	Representativeness	≤ 6°C, not frozen	S

**Notes:**

Field duplicates for soil will not be collected because of the heterogeneity of soil.

<sup>a</sup> Field QC sample analyses will be specific to the samples they are associated with.

<sup>b</sup> The laboratory will measure the temperature of the temperature blank upon receipt of the cooler. If the temperature is outside the acceptable range, the effect on the data quality of the sample results will be evaluated during data validation. No other chemical analyses will be performed on the temperature blank.

### SAP Worksheet #13—Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
IRP Site 6 Analytical Data	Jacobs Engineering <i>Final Site Inspection, Naval Fuel Support Point San Pedro, California</i> 1993	Jacobs Engineering Soil data 1992	Data were used to determine sample locations.	No limitations on final validated data.
Background Analytical Data	TriEco-Tt <i>Final Remedial Investigation Report for Installation Restoration Program Site 32, Southeast Ravine, Defense Fuel Support Point San Pedro, California</i> March 2013	TriEco-Tt Soil and groundwater data collected in support of IRP Site 32 RI 2012	Data will be used for background evaluation.	No limitations on final validated data.

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## SAP Worksheet #14—Summary of Project Tasks

This worksheet contains detailed procedures for field activities. Worksheet #21 and Attachment 3 of this SAP contain additional SOPs that will be followed during the field investigation.

### 14.1 Preparatory Activities

Prior to beginning fieldwork, the Navy RPM, Activity POC, and appropriate security and fire department personnel will be notified regarding the anticipated fieldwork and schedule. A field kickoff meeting will be conducted and may include the Navy RPM, Activity POC, a biologist, TOM, Field Manager, field personnel, PQAO, and PSHM. The primary discussion points for the meeting will include scope of work, schedule, logistics and field coordination issues, sensitive species and habitat concerns, and safe access to IRP Site 6. The appropriate subcontractors will be procured for hollow-stem auger (HSA) drilling, trenching equipment, well installation and development, chemical analysis, data validation, IDW disposal, location surveying, and geophysical survey and subsurface utility locating.

Prior to conducting field activities, field personnel will review the applicable sections of this SAP, schedule, and SSHP, and sign the Project Personnel Sign-Off Sheet (Worksheet #4). Safety considerations for proposed field tasks are discussed in the project-specific APP/SSHP (submitted under separate cover).

### 14.2 Field Logbook

Field notes will be kept in bound, weatherproof logbooks. Notes will be taken with waterproof, permanent ink. Field staff completing separate tasks will keep separate logbooks, as necessary, according to the following protocol KCH SOP 13, Preparing Field Log Books:

- Company name, address, author, activity, location, project name, TOM, and emergency contact information will be included on the inside cover of the logbook.
- All lines of all pages will be used. Any line not used will be marked through with a diagonal line, initialed, and dated. Pages not used will be marked through with a diagonal line, the author's initials, the date, and the note "Intentionally Left Blank."
- If errors are made in the logbook, a single line will be crossed through the error and the correct information entered. All corrections will be initialed and dated by the personnel performing the correction.
- Daily entries will be made chronologically and will be recorded directly in the field logbook during the work activity.
- Each page of the logbook will have the date of the work and the note taker's initials.
- The final page of each day's notes will include the date and the note taker's signature.
- Only information relevant to the subject project will be added to the logbook.
- Entries into the logbook will be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory.
- Entries must be legible and complete.

- The field notes will be copied and the copies sent to the TOM for review in a timely manner.

The following general information will be recorded in the field logbooks:

- The general scope of work to be performed each day
- Weather conditions and significant changes in the weather during the day
- Summary of onsite tailgate health and safety meetings or other meetings
- Level of personal protective equipment being used
- Instrument calibration (if applicable)
- A detailed, chronological account of activities each day
- Complete names, arrival times, departure times, roles, and affiliations of all personnel who enter the IRP site; acronyms may be used after they are established in the logbook
- Communications (visitors, phone, subcontractors, field staff) that may affect performance of the project
- Deviations from the work plan, this SAP, and the reason deviations were required
- Health and safety incidents
- Quantities of consumable equipment used, if they are to be billed to the project
- Problems encountered during the fieldwork and the CAs taken to address these problems
- Conditions that may adversely affect the work or data obtained
- Sample dates, times, and identifications (IDs)
- Field progress performed each day (samples collected, feet of drilling performed, etc.).

### **14.3 Geophysical Survey/Utility Locating**

As part of the RI, a geophysical survey will be conducted at IRP Site 6 to assess the lateral extent of disposed debris to aid in the placement of sampling locations. This effort is necessary in the northern half of IRP Site 6 where debris is expected. Because of thick vegetation and steep terrain in the northeast branch, a traditional geophysical grid is not practical. Instead, geophysical transect lines will be used. The lines will consist of approximately 4,000 feet of linear coverage with one transect parallel to each branch of the South Ravine and several transects perpendicular across the branches.

The geophysical subcontractor will also clear subsurface utilities at IRP Site 6 sample locations before intrusive field investigations begin. All clearances needed for borehole drilling will be obtained in accordance with the Navy's established procedures and requirements. All locations will be marked in the field with a wooden stake, and the sample location will be indicated on a map.

Underground Service Alert (USA) will be notified at least 2 full working days in advance of drilling or trenching activity. KCH will supplement the USA services and Navy utility information with a geophysical survey. The following geophysical technologies will be used as necessary by the geophysical subcontractor:

- **Ground-penetrating radar** can detect pipes, including gas pipes, tanks, conduits, and cables – both metallic and nonmetallic – at depths up to 10 feet. Sensitivity for both minimum object size and maximum depth detectable depends on factors such as equipment selected and soil conditions.
- **Radio frequency (RF)** detection involves inducing an RF signal in the pipe or cable and using a receiver to trace it. Some electrical and telephone lines emit RF naturally and can be detected without an induced signal. This method requires knowing where the conductive utility can be accessed to induce an RF field, if necessary.
- **Dual RF** is a modified version of RF detection using multiple frequencies to enhance sensitivity but with similar limitations to RF.
- **Ferromagnetic detectors** are metal detectors that will detect ferrous and nonferrous utilities. Sensitivity is limited (e.g., a 100-millimeter iron disk to a depth of about 1 meter or a 25-millimeter steel paper clip to a depth of about 20 centimeters).
- **Electronic markers** are emerging technologies that impart a unique electronic signature to materials such as polyethylene pipe to facilitate location and tracing after installation. These are promising for future installations but are not helpful for most existing utilities already in place.

Utility clearances provided by the geophysical subcontractor will be in writing, signed by the geophysical party conducting the clearance, and will be submitted to KCH within 24 hours of completing the utility-locating activities. Boring locations will be marked by KCH personnel prior to the USA or geophysical clearance activities.

The geophysical survey will also be used to assess debris that may not have been observed during previous investigations or included in the removal action activities. Results may be used to revise proposed trench, boring, and groundwater monitoring well locations.

#### 14.4 Surface and Subsurface Soil Sampling

Soil sampling will be conducted at IRP Site 6 to determine whether the nature and extent of debris is adequately characterized and to determine whether COPCs in soil pose an unacceptable risk to human or ecological receptors. Soil samples will be collected via hand augering, HSA drilling, and trenching.

##### Hand Augering

A hand auger will be used to clear each boring location down to approximately 5 feet bgs at IRP Site 6. The hand auger will also be used to collect soil samples from five soil borings to 10 feet bgs (Figure 10-3). The hand auger will be decontaminated prior to each sample interval consistent with the procedures discussed in Section 14.11 of this worksheet. Soil samples will be collected from 0 to 0.5 foot bgs, 4 to 6 feet bgs, and 8 to 10 feet bgs. Soil sampling depth may be adjusted in the field based on changes in lithology, soil headspace PID readings, color, odor, or at the field geologist's discretion. If refusal is encountered at an initial sample location,

additional hand auger borings will be attempted within the utility clearance footprint. Soil headspace will be screened using a PID, and readings will be recorded on the boring log. Immediately upon removal of the hand auger, a small amount of soil will be placed in a resealable plastic bag. The resealable plastic bag will be sealed, and the tip of the PID will be inserted through the wall of the sealed plastic bag to screen volatile organic concentrations. Soil samples will be collected for laboratory analysis as described in the following paragraphs.

### **Hollow-Stem Auger Drilling**

Prior to drilling at IRP Site 6, each boring location will be cleared to approximately 5 feet bgs with a decontaminated hand auger. Seven borings (KCH06-SB01 through KCH06-SB05, and KCH06-MW01 and KCH06-MW02) will be advanced using a truck-mounted HSA drill rig equipped with 8-inch-diameter augers. The locations of the borings are shown on Figure 10-3. All downhole equipment (i.e., augers and samplers) will be decontaminated before use at each boring location, consistent with the procedures discussed in Section 14.11 of this worksheet. Five of the borings (KCH06-SB01 through KCH06-SB05) will be advanced to approximately 20 feet bgs, and two of the borings (KCH06-MW01 and KCH06-MW02) will be advanced up to approximately 150 feet bgs for installation of groundwater monitoring wells. In the upper 10-foot-depth interval, soil samples will be collected from 0 to 0.5 foot bgs, 4 to 6 feet bgs, and 8 to 10 feet bgs at all seven borings. At five of the borings, soil samples will also be collected from approximately 14 to 16 feet bgs and 18 to 20 feet bgs. At the two monitoring well borings, up to 12 soil samples will be collected. From 10 to 30 feet bgs, soil samples will be collected in 5-foot sample intervals. And from 30 feet to groundwater, soil samples will be collected at 20-foot intervals.

Soil samples collected during HSA drilling will be collected using a California modified split-spoon sampler lined with stainless steel sleeves. HSA cuttings will be visually observed by the field geologist with observations recorded on a KCH Boring Log (Attachment 4) for each boring. The soils will be described consistent with the American Society for Testing and Materials International (ASTM) Method D 2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)* and KCH SOP 5, Logging of Soil Borings. Fill or soil color will be based on the Munsell Color System. Soils will be screened using a PID, and PID readings will be recorded on the KCH Boring Log. Immediately upon removal of the sample liner from the split-spoon sampler, a small volume of soil will be placed in a resealable plastic bag. The resealable plastic bag will be sealed, allowed to equilibrate for 5 to 10 minutes, and then the tip of the PID will be inserted through the wall of the sealed plastic bag to screen volatile organic concentrations in the soil vapor headspace. Fill or soil samples will be collected consistent with procedures described in the Soil Sampling section of this worksheet.

### **Trenching**

Twelve trenches will be excavated using an all-terrain backhoe with extendable bucket boom at IRP Site 6 (Figure 10-3). The backhoe bucket will be decontaminated prior to excavating consistent with the procedures discussed in Section 14.11 of this worksheet. The trenches will extend to an estimated depth of 10 feet bgs, 2 to 3 feet wide (contingent upon soil conditions to keep an open, stable trench), and will be approximately 10 feet long. The excavated soil and debris removed from the trench will be placed on plastic sheeting at least 5 feet away from the edge of the trench to avoid caving.

Each bucket of material removed from the trench will be visually observed by the field geologist, and a KCH Trench Log (Attachment 5) will be maintained. The KCH Trench Log will include a description of the physical nature of the rubble and debris encountered. The depth at which native soil (i.e., silt, sand, clay, gravel, rock) is encountered (as deep as 10 feet bgs or deeper as allowed by the capability of the excavating equipment) will be noted on the KCH Trench Log, and the native soil will be described in accordance with the ASTM Method D 2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)* and consistent with KCH SOP 5, Logging of Soil Borings. Soil color will be based on the Munsell Color System. Excavated trench material will be screened using a PID, and readings will be recorded on the KCH Trench Log. Soil headspace PID readings for trench soils will be performed similar to the procedure discussed in the HSA Drilling section that precedes this section.

Digital photographs will be taken showing the location of each trench within IRP Site 6, the trench walls (both sides of the trench, if possible), the bucket samples that are sampled, and unusual conditions. These photographs will document the nature of the surface and subsurface soil, the debris, and the changing conditions or lithology. A description of each digital photograph will be recorded in the field logbook.

An average of three soil samples will be collected at each of the 12 trenches, for a total of up to 36 samples, within IRP Site 6. Sample collection depth will depend of site conditions; however, depths will include both surface (0 to 0.5 foot bgs) and subsurface sample locations. The soil sample locations will be biased to characterize the rubble debris. Soil samples will be collected from the excavator bucket using decontaminated or disposable scoops or trowels, as necessary. Soil sampling will be completed as described in the Hand Augering and Soil Sampling sections of this worksheet.

When sample collection is complete, the trench will be backfilled with the original material unless there are visible signs of contamination. Backfilled soil will be compacted with the backhoe bucket. The plastic sheeting will be rinsed as needed and disposed of as nonhazardous waste. If there are visible signs of contamination, soil or debris will be placed in a roll-off bin for disposal as outlined in Section 14.10.

Trench locations were chosen based on how well they allow mechanized equipment to access these locations that require delineation of COPCs. Actual trench locations will be based in part on the results of the geophysical survey and visual observations of potential waste or debris. Prior to trenching, KCH field personnel will inspect the ground surface and overhead space to identify potential obstructions that could cause a safety hazard (e.g., subsurface utilities, overhead power lines, or trees). Site obstructions will be noted in the field so that KCH can set up the excavator at an alternate location, if needed, to mitigate potential safety hazards. Samples collected from trenches will be taken from the backhoe bucket; field personnel will not enter the trench.

At each trench, thickness of the soil cover over the waste and soil classification will be documented. The character and content of encountered waste, amount of soil mixed with the waste, and the bottom depth of the waste, will also be documented. Soil, waste, and other significant features (e.g., burn ash) will be described and photographed, with observation details recorded in the field logbook.

## Soil Sampling

Soil samples will be collected directly from the hand auger, a California modified split-spoon sampler lined with stainless steel sleeves, or from the excavator bucket. If samples require VOC or TPH-p analyses (based on visible contamination, odors, or PID readings in excess of 5 ppm), the soil sample will be collected from the end of the hand auger or stainless steel liner, at the appropriate depth, immediately after removal.

Samples to be analyzed for VOCs and TPH-p will be collected using an EnCore (or equivalent) sampler for each sampling method. The EnCore (or equivalent) sampler is a dedicated, single-use system designed to collect, store, and deliver approximately 5 grams of soil. The system consists of a plunger, a cap that seals the plunger with an O-ring, and a T-handle to aid in sample collection. The plunger will be placed into the T-handle and inserted into the soil. The volumetric sampling device (i.e., soil plunger) will then be immediately sealed with the cap and sent to the laboratory for analysis. After the EnCore (or equivalent) samples are collected, the remaining contents of the hand auger, the stainless steel liner, or the material from the excavator bucket will be emptied into a decontaminated, stainless steel mixing bowl. Rocks, sticks, and organic material will be removed from the mixing bowl. The remaining soil in the bowl will be field-homogenized by thoroughly mixing the soil with a decontaminated, stainless steel spoon or spatula, and the remaining laboratory-supplied sample jars will be filled for submittal to the analytical laboratory.

Soil samples will be submitted to a DoD ELAP-accredited analytical laboratory for the following analyses:

- SVOCs by USEPA 8270C.
- PAHs by USEPA 8270C SIM.
- OCPs by USEPA 8081A.
- PCBs by USEPA 8082.
- Metals by USEPA 6020A.
- Mercury by USEPA 7471A.
- Hexavalent chromium by USEPA 7199.
- VOCs by USEPA 8260B; contingency analysis analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- TPH-p by USEPA 8015B; contingency analysis analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- TPH-e by USEPA 8015B; contingency analysis analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible contamination, odors, or PID readings in excess of 5 ppm.
- Dioxins/furans by USEPA 8290A; contingency analysis analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations based on the presence of visible evidence of burning (i.e., soot, ash, or other combustion by-products).

Soil will be logged consistent with ASTM Method D 2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)*. Soil color will be based on the Munsell system. Soil boring logs will be completed on KCH Soil Boring Log separate from the field logbook. An example of a KCH Soil Boring Log is included as Attachment 4. A PID will be used to screen the soil for volatile organic concentrations immediately upon retrieval, and the PID readings will be recorded on the KCH Soil Boring Log. Calibration information for the PID is described in Worksheet #22. Calibration information will be recorded in the field logbook.

Each hand auger borehole will be backfilled by pouring bentonite chips into the borehole to within a few inches of the surface and hydrating. Each HSA borehole will be backfilled with cement grout with 5 percent bentonite (pumped into the borehole from the bottom up with a tremie pipe) to within a few inches of the surface. All boreholes will be patched with native soil to match existing surface conditions.

Before the onset of hand augering, drilling, and trenching; between borehole locations; and before leaving each sample location, nondisposable sampling equipment (e.g., the hand auger, drill stem, and core barrel) will be decontaminated as described in Section 14.11. Excess soil cuttings and decontamination water will be containerized in United States Department of Transportation (DOT)-approved 55-gallon drums and stored in a secure storage location designated by the Navy RPM.

## 14.5 Monitoring Well Installation

Two monitoring wells (KCH06-MW01 and KCH06-MW02) will be installed at IRP Site 6 (Figure 10-3) to evaluate the following: whether COPCs in groundwater have been adequately characterized, whether the groundwater beneath IRP Site 6 contains COPCs, and if so, whether the COPCs pose an unacceptable risk to human receptors.

Well boreholes will be advanced using HSA drilling. Soil samples will be collected every 5 feet with a California modified split-spoon sampler and will be logged by a California-registered geologist using the Unified Soil Classification System and recorded on a KCH Boring Log. The first occurrence of groundwater (i.e. saturated sediments) will be recorded on the KCH Boring Log. One monitoring well will be installed into the uppermost saturated interval at each of the two proposed monitoring well locations.

Monitoring wells will be installed and constructed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981 and 1990). Each monitoring well will be constructed of 4-inch-diameter, Schedule 40, flush-jointed polyvinyl chloride (PVC) casing and factory-slotted well screen. Monitoring wells will be completed in a 10-inch-outer-diameter borehole. Wells will be constructed within the first encountered groundwater. The total well depth is expected not to exceed 150 feet bgs. A 30-foot-long well screen will be used (10 feet above and 20 feet below the water table) to accommodate changing regional water levels, as a result of the current drought conditions. The well screen will have 0.020-inch slotted openings (four cuts per round). A monitoring well construction diagram is presented on Figure 14-1.

A filter pack of clean #3 Monterey sand will be poured from ground surface into the annular space between the auger flights and the well casing/well screen. If the well is constructed in an open borehole, then a tremie pipe will be used to emplace the filter pack. The filter pack will extend a minimum of 3 feet above the top of the screen. A 10-foot-thick layer (minimum) of

hydrated bentonite (as chips or pellets) will be placed on top of the filter pack and allowed to fully hydrate before the remaining annular space is filled with cement-bentonite grout.

A small notch will be cut on the northern side of the top of the PVC casing to be used as a reference for future water level measurements. The well casing will be completed approximately 2 feet above ground surface. The aboveground well completions will use an outer 5-foot-long protective steel surface casing with a locking cap, extending approximately 2.5 feet above ground surface and surrounded by three or four protective steel bollards. A cement well pad approximately 3 feet square and a minimum of 4 inches thick that slopes away from the protective outer steel casing will be used. The well name will be clearly identified on the well pad or protective outer casing.

## 14.6 Monitoring Well Development

New monitoring wells will be developed by a drilling subcontractor prior to groundwater sampling. Monitoring well development will be performed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981 and 1990), using the surge-and-bail method followed by pumping, as discussed in the following paragraph.

Groundwater monitoring wells will be developed by the surge-and-bail method followed by pumping to improve hydraulic communication between the geologic formation and the well, to remove suspended fines, and to result in a monitoring well that will produce sediment-free, low turbidity groundwater samples. For each well, the development procedure will first include measuring the depth to groundwater and calculating the volume of water within the well casing. Subsequently, each of the wells will be surged using a surge block within approximately 5-foot intervals of the saturated portion of the screened interval for approximately 10 minutes for each 5-foot interval. Next, the wells will be bailed or pumped to remove a minimum of three casing volumes of groundwater or until the pumped groundwater is free of visible turbidity and turbidity is less than 10 nephelometric turbidity units (NTUs). Water quality parameters, including hydrogen ion concentration (pH), temperature, turbidity, electrical conductivity (EC), dissolved oxygen (DO), and oxidation-reduction potential (ORP) will be monitored until they stabilize (Step 16 of Section 14.7) or until extracted groundwater becomes visually clear and turbidity is less than 10 NTUs, at which time development will be considered complete. The purge volumes, indicator parameters, and estimated recharge rates will be recorded in the field logbook during well development.

Development water will be transferred to DOT-approved 55-gallon drums. The drums will be sealed, labeled, and stored in a secure storage location designated by the Navy RPM.

## 14.7 Groundwater Sampling

The goal of groundwater sampling at IRP Site 6 is to evaluate whether COPCs have been adequately characterized in groundwater at IRP Site 6 and whether they pose an unacceptable risk to human receptors. New monitoring wells (KCH06-MW01 and KCH06-MW02) and existing monitoring wells (SB-10 and B-21) (Figure 10-3) will be monitored and sampled as indicated in the following discussion.

Groundwater monitoring will begin a minimum of 72 hours after the completion of new well development or redevelopment of existing wells. At each of the groundwater monitoring wells (Worksheet #18), groundwater samples will be collected consistent with the USEPA guidance

*Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (Puls and Barcelona, 1996).

The low-flow guidance, along with this SAP, will be reviewed by sampling personnel prior to sampling activities. The monitoring wells will not have dedicated pumping systems, so temporary bladder pump sampling systems will be used for sample collection. Details of the groundwater sampling procedure are as follows:

1. Upon removal of the PVC cap from each well, a PID will be used to screen for organic vapors on the inside of the well head casing. This PID reading will be recorded on the KCH Low-Flow Groundwater Sampling Sheet (Attachment 6). A decontaminated water level meter will be used to measure the depth to groundwater (feet below top of casing) and the depth to the bottom of the well (feet below top of casing) to check for accumulated sediment or other obstructions at each well prior to sampling. Measurements will be made from the northern side of the casing (unless a mark is present on the casing) and recorded on the KCH Low-Flow Groundwater Sampling Sheet. The water level meter will be decontaminated between well locations (see Section 14.11).
2. Prior to beginning sampling activities and between sampling locations, the bladder pump will be decontaminated.
3. A new, clean, disposable Teflon bladder assembly will be installed in the pump prior to sampling at each location.
4. New, clean bailing string will be attached to the pump assembly at each location according to manufacturer's instructions.
5. New, clean air compressor tubing will be attached to the pump assembly at each location according to manufacturer's instructions.
6. New, clean purge tubing (Teflon or Teflon-lined polyethylene tubing is preferred when sampling for organic compounds) will be attached to the pump assembly at each location according to manufacturer's instructions.
7. Once string and tubing are attached to the pump assembly, the pump will be slowly lowered into the monitoring well using the bailing string to bear the weight of the pump, while keeping a small amount of slack in the purge tubing so the tubing does not detach from the pump assembly. **THE TUBING SHALL NOT BE USED TO LOWER THE PUMP INTO THE MONITORING WELL.**
8. Once the pump intake is lowered to approximately the middle of the screened interval, the bailer string will be tied off to hold the pump at that depth.
9. The air compressor tubing will be connected to the air compressor or controller according to manufacturer's instructions.
10. The purge tubing will be connected to the flow-through cell of the multi-parameter meter (e.g., YSI-556) that will measure pH, temperature, DO, EC, turbidity, and ORP during purging. Calibration information for the multi-parameter meter is described in Worksheet #22. Calibration information will be recorded in the field logbook.
11. The flow-through cell will be set up to expel purge water into 5-gallon buckets.

12. The water level meter will be lowered into the well to continually measure depth-to-water during purging.
13. The air compressor or controller will be attached to the power source (e.g., car battery or generator). The exhaust from the compressor will be directed downwind of sampling activities.
14. When ready to begin sampling, the air compressor or controller will be turned on and the flow rate will be adjusted to approximately 0.1 to 0.5 liter per minute (L/min). If the drawdown begins to exceed 0.3 foot, the purge rate will be slowed. If the drawdown still continues to exceed 0.3 foot, the information will be recorded on the KCH Low-Flow Groundwater Sampling Sheet.
15. During purging, field measurements will be recorded for depth-to-water, drawdown, pH, temperature, DO, EC, ORP, and turbidity approximately every 3 minutes on the KCH Low-Flow Groundwater Sampling Sheet.
16. After three successive readings of  $\pm 0.1$  for pH,  $\pm 3$  percent for conductivity, and  $\pm 10$  percent for turbidity (if above 10 NTUs) and DO, the monitoring well will be considered to be stabilized and ready for collection of representative groundwater samples. DO and turbidity usually require the longest time for stabilization (Puls and Barcelona, 1996). The turbidity reading needs to stabilize at a value below 10 NTUs (USEPA, 2002b).
17. The flow-through cell for the multi-parameter meter will be disconnected.
18. The flow rate will be adjusted as appropriate for sample collection (less than 0.5 L/min, or 100 milliliters per minute for volatile organic analyses).
19. Sample collection will begin with sample bottles for volatile analyses (VOCs and TPH-p), followed by TPH-e, then the samples for the less volatile constituents.
20. The aliquots for dissolved metals and hexavalent chromium will be filtered in the field using a disposable 0.45-micron filter. The aliquot for dissolved metals will be collected in a method-specific container with nitric acid. The aliquot for hexavalent chromium will be placed in a method-specific container with ammonium hydroxide and ammonium sulfate buffer solution. The pH of the sample will be checked with pH paper. If the pH of the sample is not between 9.0 and 9.5, additional ammonium buffer solution will be added to the sample. Samples will be packaged and transported to the analytical laboratory.
21. Once sampling is complete, the air compressor or controller will be turned off, and the power source will be turned off and disconnected.
22. Sample handling according to COC protocol will begin immediately.
23. The pump will be removed from the monitoring well by pulling up the bailing string. The discharge tubing and pump will be placed onto clean plastic sheeting.
24. Once the pump is retrieved to surface, tubing and string will be removed.
25. The pump will be disassembled according to manufacturer's instructions. The bladder will be removed and disposed of, and then the decontamination of the pump will begin.

Groundwater samples will be submitted to a DoD ELAP-accredited analytical laboratory for the analyses listed on Worksheet #18 and as follows:

- VOCs by USEPA 8260B
- TPH-p by USEPA 8015B
- TPH-e by USEPA 8015B
- SVOCs by USEPA 8270C
- PAHs by USEPA 8270C SIM
- OCPs by USEPA 8081A
- PCBs by USEPA 8082
- Total and Dissolved Metals by USEPA 6020A
- Total and Dissolved Mercury by USEPA 7470A
- Hexavalent chromium by USEPA 218.6

Groundwater samples will be handled consistent with KCH SOP 16, Chain of Custody. Waterproof sample labels will be filled out completely and attached to each sample container. Samples will be placed in resealable plastic bags and stored in a cooler with ice. Bubblewrap may be used to wrap and protect glass containers. The COC documentation will be completed as samples are collected. At the end of the day, COC documentation will be verified against sample labels in the associated cooler by designated field personnel and noted in the field logbook. Each day, samples will be relinquished either to a courier provided by the analytical laboratory or by overnight carrier consistent with COC protocol.

Purge and decontamination water will be transferred to DOT-approved 55-gallon drums. The drums will be sealed, labeled, and stored in a secured location designated by the Navy RPM.

## **14.8 Field Equipment Decontamination**

All nondedicated sampling and drilling equipment will be decontaminated between sampling locations using the following three-step process, consistent with KCH SOP 11, Decontamination of Personnel and Equipment:

1. Wash equipment with tap water and Liquinox (or equivalent) solution using brushes or a high pressure steam cleaner.
2. Rinse with tap water.
3. Rinse with distilled water, then air dry on a clean, uncontaminated surface (such as aluminum foil).

The drilling subcontractor may use a combination of the three-step process and high-pressure steam cleaning to complete decontamination. Decontamination water will be transferred to DOT-approved 55-gallon drums. The drums will be sealed, labeled, and stored in a secured location designated by the Navy RPM.

## **14.9 Location Surveying**

All new monitoring well and borehole locations will be surveyed by a professional land surveyor, licensed by the State of California. Both ends of each new trench will be surveyed. The surveyor will provide the elevation at backfilled ground surface to a precision of 0.01 foot and its location to a precision of plus or minus 0.1 foot horizontally, based on the borehole

center. The elevations will be surveyed relative to the 1988 National Geodetic Vertical Datum. The borehole and monitoring well locations will be surveyed using the 1983 North American Datum State Plane Coordinate System, California, Zone 5. Vertical coordinates will be reported as feet relative to msl.

## 14.10 Management of IDW

IDW generated during this investigation will include soil cuttings from hand augering and HSA drilling, and trenching; decontamination water; and purge water. All soil and water will be placed in DOT-approved 55-gallon drums or roll-off bins, sealed, labeled (drums), and stored in a secure location designated by the Navy RPM until disposal can be secured at an offsite treatment or recycling facility. Sampling and analysis of IDW will be required for waste disposal profiling. Waste containment, labeling, sampling, and disposal will all be handled as part of IDW management. DFSP San Pedro will provide a secure waste storage facility. KCH will provide leak protection features (e.g., underlying leak proof floor or plastic sheet surrounded by a berm to contain leaks or rainfall runoff) for the temporary accumulation of IDW. Authorized DFSP San Pedro personnel will sign waste manifests. IDW is anticipated to be profiled as nonhazardous based on prior experience.

## 14.11 Data Validation and Evaluation

Data validation will be conducted by an independent third-party data validation subcontractor consistent with NAVFAC Southwest Environmental Work Instruction (EWI) No. 1, *Data Validation Guidelines for Chemical Analysis of Environmental Samples* (NAVFAC Southwest, 2001); *USEPA National Functional Guidelines for Organic Data Review* (USEPA, 2014a); *USEPA National Functional Guidelines for Inorganic Superfund Data Review* (USEPA, 2014b); and *USEPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (USEPA, 2011). All (100 percent) of the analytical data will be validated by a third-party data validation subcontractor, with 10 percent full (Level IV) validation and 90 percent standard (Level III) validation.

Full (Level IV) data validation will follow the USEPA criteria set forth in the functional guidelines for organic, inorganic, and dioxin/furan data review (USEPA, 2014a, 2014b, and 2011, respectively). These functional guidelines apply to analytical data packages that include the raw data (e.g., spectra and chromatograms), backup documentation for calibration standards, analysis run logs, laboratory quality control, dilution factors, and other types of information. This additional information is used in the full (Level IV) data validation process for checking calculations of quantified analytical data. Calculations are checked for field and laboratory QC samples (e.g., matrix spike/matrix spike duplicate [MS/MSD] and laboratory control sample [LCS] results) and routine field samples (including field duplicates, source blanks, equipment rinsate blanks, and trip blanks). To be sure that detection limit and data values are appropriate, an evaluation is made of instrument performance, method of calibration, and the original data for calibration standards.

Under the standard (Level III) data validation effort, the data values for routine samples and field and laboratory QC samples are generally anticipated to be correctly reported by the laboratory. Data quality is assessed by comparing the QC parameters listed above to the appropriate criteria (or limits) as specified in this SAP, by functional guidelines, or by method-specific requirements (e.g., SW-846). If calculations for quantitation are verified, it is

done on a limited basis and may require raw data in addition to the standard data forms that are normally in a data package.

The data validation reports will include applicable data qualifiers for each analyte result and changes to the reporting limits as a result of required sample dilutions to bring the sample within the linear concentration calibration range of the analytical method or to minimize matrix effects. If samples have been analyzed at multiple dilutions, then each reported analyte value will use the lowest sample dilution consistent with the linear concentration range of the analytical method

## 14.12 Data Management

Data management will begin upon collection of field measurements, which will be recorded in the investigation-specific field logbook and retained in the project files, to the final submittal of the analytical data that are checked for accuracy through the data validation process. Throughout the data life cycle, the project team and project subcontractors (i.e., laboratory and data validation) will be responsible for performing data verification so that the data are complete, correct, and compliant with project objectives and contractual requirements.

Analytical data will be provided by the analytical laboratory in hard-copy and electronic KCH-required formats. If the electronic data have successfully passed the data checker, the laboratory will provide the analytical data in the contractually required EDD format, which will then be loaded to the KCH data management system for further data verification and validation. The data will not be released to the project team or Navy until the data are final with data validation qualifiers applied. Once the data are deemed final and complete, the data will be prepared in the required NEDD format and uploaded to NIRIS, consistent with NAVFAC Southwest's EWI No. 6, *Environmental Data Management and Required Electronic Delivery Standards* (NAVFAC Southwest, 2005).

## 14.13 Reporting

Final, validated, soil and groundwater analytical data collected during this investigation will be presented in the RI report for IRP Site 6. The RI report will include the final, validated, analytical data collected as part of this investigation. The RI report will include data reduction, tabulation and analysis, report preparation, QC checks, and response to comments (RTCs). The report will include a description of field activities, CSM, analytical results, surface and subsurface soil and debris conditions, the occurrence of groundwater, the nature and extent of COPCs in soil and groundwater, and recommendations for further investigation or no further action. The report will also present the findings of a HHRA and ERA, including Step 3A, risk refinement, for IRP Site 6.

The primary objective of the RI report is to investigate the nature and extent of COPCs at IRP Site 6 and to assess whether the COPCs at IRP Site 6 pose an unacceptable risk to human health or the environment. If a data review indicates that further action is required, then additional sampling will be recommended. Attachments to the draft and final versions of the RI report will include field sampling reports with trench and boring logs, analytical data, validation reports, aerial photographs (if available), photographs of trenches and subsurface soil samples, risk assessment results, and other supporting data from previous activities. The report will be submitted as preliminary draft, draft, and final versions. The Navy will be provided with each version for review and comment, and documents will be reviewed and

approved by the Navy prior to submittal to regulatory agencies. RTCs will be prepared for each comment set received. The RTCs will be used at each review step to facilitate approval of comment responses.

## SAP Worksheet #15a—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Acetone	67-64-1	14,000	USEPA Tap Water RSL	10	10	5.0	2.6
Benzene	71-43-2	0.45 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Bromodichloromethane	75-27-4	0.13 <sup>c</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Bromoform	75-25-2	3.3	USEPA Tap Water RSL	1.0	1.0	0.3	0.15
Bromomethane	74-83-9	7.5	USEPA Tap Water RSL	1.0	1.0	0.3	0.16
2-Butanone	78-93-3	5,600	USEPA Tap Water RSL	10	10	5.0	2.0
Carbon disulfide	75-15-0	810	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Carbon tetrachloride	56-23-5	0.11 <sup>c</sup>	DTSC Tap Water Screening Level	1.0	1.0	0.2	0.10
Chlorobenzene	108-90-7	70	State of California MCL	1.0	1.0	0.2	0.10
Chloroethane	75-00-3	4.8	DTSC Tap Water Screening Level	1.0	1.0	0.3	0.27
Chloroform	67-66-3	0.22 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Chloromethane	74-87-3	190	USEPA Tap Water RSL	1.0	1.0	0.3	0.15
Dibromochloromethane	124-48-1	0.17 <sup>c</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,2-Dibromoethane	106-93-4	0.0075 <sup>d</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,2-Dichlorobenzene	95-50-1	300	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,3-Dichlorobenzene	541-73-1	180	DTSC Tap Water Screening Level	1.0	1.0	0.2	0.11
1,4-Dichlorobenzene	106-46-7	0.48 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Dichlorodifluoromethane (Freon 12)	75-71-8	200	USEPA Tap Water RSL	1.0	1.0	0.3	0.15
1,1-Dichloroethane	75-34-3	2.7	USEPA Tap Water RSL	1.0	1.0	0.2	0.10

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Matrix: Water

Analytical Group: VOCs – USEPA Method 8260B

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,2-Dichloroethane	107-06-2	0.17 <sup>c</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,1-Dichloroethene	75-35-4	6.0	State of California MCL	1.0	1.0	0.2	0.10
cis-1,2-Dichloroethene	156-59-2	6.0	State of California MCL	1.0	1.0	0.2	0.10
trans-1,2-Dichloroethene	156-60-5	10	State of California MCL	1.0	1.0	0.2	0.10
1,2-Dichloropropane	78-87-5	0.44 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,1-Dichloropropene	563-58-6	0.47 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.2	0.10
cis-1,3-Dichloropropene	10061-01-5	0.47 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.2	0.10
trans-1,3-Dichloropropene	10061-02-6	0.47 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.2	0.11
Ethylbenzene	100-41-4	1.5	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Hexachlorobutadiene	87-68-3	0.3 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.3	0.22
2-Hexanone	591-78-6	38	USEPA Tap Water RSL	10	10	5.0	2.3
Methylene chloride	75-09-2	3.6	DTSC Tap Water Screening Level	2.0	2.0	1.0	0.5
4-Methyl-2-pentanone	108-10-1	1,200	USEPA Tap Water RSL	10	10	5.0	2.1
Methyl tertiary butyl ether	1634-04-4	13	State of California MCL	1.0	1.0	0.2	0.13
Naphthalene	91-20-3	0.17 <sup>d</sup>	USEPA Tap Water RSL	2.0	2.0	1.0	0.5
1,1,2,2-Tetrachloroethane	79-34-5	0.076 <sup>d</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.11
Tetrachloroethene	127-18-4	0.19 <sup>c</sup>	DTSC Tap Water Screening Level	1.0	1.0	0.2	0.15
Tertiary butyl alcohol	75-65-0	24,000	USEPA Tap Water RSL <sup>e</sup>	10	10	5.0	2.5
Styrene	100-42-5	100	California and Federal MCL	1.0	1.0	0.5	0.25

## SAP Worksheet #15a—Reference Limits and Evaluation Table

Matrix: Water

Analytical Group: VOCs – USEPA Method 8260B

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Toluene	108-88-3	150	State of California MCL	1.0	1.0	0.2	0.10
1,2,4-Trichlorobenzene	120-82-1	1.1	USEPA Tap Water RSL	1.0	1.0	0.3	0.15
1,1,1-Trichloroethane	71-55-6	200	California and Federal MCL	1.0	1.0	0.2	0.10
1,1,2-Trichloroethane	79-00-5	0.28 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Trichloroethene	79-01-6	0.49 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Trichlorofluoromethane (Freon 11)	75-69-4	150	State of California MCL	1.0	1.0	0.3	0.15
1,2,4-Trimethylbenzene	95-63-6	15	USEPA Tap Water RSL	1.0	1.0	0.2	0.11
1,3,5-Trimethylbenzene	108-67-8	120	USEPA Tap Water RSL	1.0	1.0	0.2	0.13
m,p-Xylenes	108-38-3/106-42-2	190	USEPA Tap Water RSL <sup>e</sup>	2.0	2.0	0.4	0.21
o-Xylene	95-47-6	190	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,1,1,2-Tetrachloroethane	630-20-6	0.57 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
1,2,3-Trichlorobenzene	87-61-6	7.0	USEPA Tap Water RSL	1.0	1.0	0.3	0.15
1,2,3-Trichloropropane	96-18-4	0.00075 <sup>d</sup>	USEPA Tap Water RSL	2.0	2.0	0.5	0.25
1,2-Dibromo-3-chloropropane	96-12-8	0.0086 <sup>d</sup>	USEPA Tap Water RSL	2.0	2.0	0.5	0.25
1,3-Dichloropropane	142-28-9	110	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
2,2-Dichloropropane	594-20-7	0.44 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.2	0.16
2-Chlorotoluene	95-49-8	240	USEPA Tap Water RSL	1.0	1.0	0.2	0.12
4-Chlorotoluene	106-43-4	250	USEPA Tap Water RSL	1.0	1.0	0.2	0.11
Bromobenzene	108-86-1	62	USEPA Tap Water RSL	1.0	1.0	0.2	0.10

## SAP Worksheet #15a—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Bromochloromethane	74-97-5	83	USEPA Tap Water RSL	1.0	1.0	0.2	0.11
Dibromomethane	74-95-3	8.0	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
Isopropylbenzene	98-82-8	450	USEPA Tap Water RSL	1.0	1.0	0.2	0.10
n-Butylbenzene	104-51-8	290	DTSC Tap Water Screening Level	1.0	1.0	0.2	0.17
n-Propylbenzene	103-65-1	660	USEPA Tap Water RSL	1.0	1.0	0.2	0.13
p-Isopropyltoluene	99-87-6	450	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.2	0.14
sec-Butylbenzene	135-98-8	590	DTSC Tap Water Screening Value	1.0	1.0	0.2	0.13
tert-Butylbenzene	98-06-6	690	USEPA Tap Water RSL	1.0	1.0	0.2	0.13
Vinyl chloride	75-01-4	0.019 <sup>d</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.12

## SAP Worksheet #15a—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal (USEPA, 2015) and State of California MCL (CDPH, 2014), the USEPA tap water RSL (USEPA, 2015), and DTSC-recommended tap water screening levels (DTSC, 2015).

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>c</sup> The LOQ and LOD do not meet the PAL; however, the DL is sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>d</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the PAL, but data evaluation will be based on reported concentrations above the DL.

<sup>e</sup> The following surrogate chemicals were used:

- 1,3-Dichloropropene for 1,1-dichloropropene, cis-1,3-dichloropropene, and trans-1,3-dichloropropene
- Sec-butyl alcohol for tertiary butyl alcohol
- 1,2-Dichloropropane for 2,2-dichloropropane
- m-Xylenes for m,p-xylenes
- Cumene (isopropylbenzene) for p-isopropyltoluene

µg/L = microgram per liter

CAS = Chemical Abstracts Service

LOD = limit of detection

MCL = maximum contaminant level

QL = quantitation limit

RSL = USEPA Regional Screening Level

## SAP Worksheet #15b—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Acetone	67-64-1	61,000,000	USEPA Residential Soil RSL	10	10	5.0	3.1
Benzene	71-43-2	330	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
Bromodichloromethane	75-27-4	290	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Bromoform	75-25-2	19,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
Bromomethane	74-83-9	6,800	USEPA Residential Soil RSL	10	10	2.0	1.8
2-Butanone	78-93-3	27,000,000	USEPA Residential Soil RSL	10	10	5.0	2.5
Carbon disulfide	75-15-0	770,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Carbon tetrachloride	56-23-5	99	DTSC Soil Screening Level	5.0	5.0	1.0	0.54
Chlorobenzene	108-90-7	280,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Chloroethane	75-00-3	3,100	DTSC Soil Screening Level	5.0	5.0	2.0	1.3
Chloroform	67-66-3	320	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Chloromethane	74-87-3	110,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
Dibromochloromethane	124-48-1	750	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,2-Dibromoethane	106-93-4	36	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,2-Dichlorobenzene	95-50-1	1,800,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,3-Dichlorobenzene	541-73-1	240,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.52
1,4-Dichlorobenzene	106-46-7	2,600	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Dichlorodifluoromethane (Freon 12)	75-71-8	87,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.2
1,1-Dichloroethane	75-34-3	3,600	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,2-Dichloroethane	107-06-2	460	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,1-Dichloroethene	75-35-4	230,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5

## SAP Worksheet #15b—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
cis-1,2-Dichloroethene	156-59-2	190,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
trans-1,2-Dichloroethene	156-60-5	190,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
1,2-Dichloropropane	78-87-5	1,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,1-Dichloropropene	563-58-6	580	DTSC Soil Screening Level <sup>b</sup>	5.0	5.0	1.0	0.5
cis-1,3-Dichloropropene	10061-01-5	580	DTSC Soil Screening Level <sup>b</sup>	5.0	5.0	1.0	0.5
trans-1,3-Dichloropropene	10061-02-6	580	DTSC Soil Screening Level <sup>b</sup>	5.0	5.0	1.0	0.5
Ethylbenzene	100-41-4	5,800	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Hexachlorobutadiene	87-68-3	1,200	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
2-Hexanone	591-78-6	200,000	USEPA Residential Soil RSL	10	10	5.0	2.9
Methylene chloride	75-09-2	5,500	DTSC Soil Screening Level	10	10	2.0	1.0
4-Methyl-2-pentanone	108-10-1	5,300,000	USEPA Residential Soil RSL	10	10	5.0	2.8
Methyl tertiary butyl ether	1634-04-4	47,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Naphthalene	91-20-3	3,800	USEPA Residential Soil RSL	10	10	2.0	1.0
1,1,2,2-Tetrachloroethane	79-34-5	600	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Tetrachloroethene	127-18-4	600	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
Tertiary butyl alcohol	75-65-0	130,000,000	USEPA Residential Soil RSL <sup>b</sup>	20	20	10	9.2
Styrene	100-42-5	6,000,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Toluene	108-88-3	1,100,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
1,2,4-Trichlorobenzene	120-82-1	24,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
1,1,1-Trichloroethane	71-55-6	1,700,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
1,1,2-Trichloroethane	79-00-5	1,100	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Trichloroethene	79-01-6	940	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5

## SAP Worksheet #15b—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Trichlorofluoromethane (Freon 11)	75-69-4	730,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.1
1,2,4-Trimethylbenzene	95-63-6	58,000	USEPA Residential Soil RSL	5.0	5.0	2.0	0.6
1,3,5-Trimethylbenzene	108-67-8	210,000	DTSC Soil Screening Level	5.0	5.0	2.0	0.6
m,p-Xylenes	108-38-3/106-42-2	550,000	USEPA Residential Soil RSL	10	10	2.0	1.0
o-Xylene	95-47-6	650,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,1,1,2-Tetrachloroethane	630-20-6	2,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
1,2,3-Trichlorobenzene	87-61-6	63,000	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
1,2,3-Trichloropropane	96-18-4	5.1	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
1,2-Dibromo-3-chloropropane	96-12-8	5.3	USEPA Residential Soil RSL	5.0	5.0	2.0	1.0
1,3-Dichloropropane	142-28-9	420,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.5
2,2-Dichloropropane	594-20-7	1,000	USEPA Residential Soil RSL <sup>b</sup>	5.0	5.0	2.0	1.0
2-Chlorotoluene	95-49-8	480,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.82
4-Chlorotoluene	106-43-4	440,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.67
Bromobenzene	108-86-1	290,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Bromochloromethane	74-97-5	150,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Dibromomethane	74-95-3	23,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.5
Isopropylbenzene	98-82-8	1,900,000	USEPA Residential Soil RSL	5.0	5.0	2.0	0.64
n-Butylbenzene	104-51-8	1,200,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.7
n-Propylbenzene	103-65-1	3,800,000	USEPA Residential Soil RSL	5.0	5.0	1.0	0.65
p-Isopropyltoluene	99-87-6	1,900,000	USEPA Residential Soil RSL <sup>b</sup>	5.0	5.0	1.0	0.62
sec-Butylbenzene	135-98-8	2,200,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.67
tert-Butylbenzene	98-06-6	2,200,000	DTSC Soil Screening Level	5.0	5.0	1.0	0.62

## SAP Worksheet #15b—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: VOCs – USEPA Method 8260B**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Vinyl chloride	75-01-4	59	USEPA Residential Soil RSL	5.0	5.0	2.0	1.4

Notes:

<sup>a</sup> The PAL is the more conservative value between the USEPA residential soil RSL (USEPA, 2015) and the DTSC soil screening levels (DTSC, 2015).

<sup>b</sup> The following surrogate chemicals were used:

1,3-Dichloropropene for 1,1-dichloropropene, cis-1,3-dichloropropene, and trans-1,3-dichloropropene

Sec-butyl alcohol for tertiary butyl alcohol

1,2-Dichloropropane for 2,2-dichloropropane

Isopropylbenzene for p-isopropyltoluene

µg/kg = microgram per kilogram

## SAP Worksheet #15c—Reference Limits and Evaluation Table

**Matrix: Water**  
**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Acenaphthene	83-32-9	530	USEPA Tap Water RSL	10	10	5.0	2.5
Acenaphthylene	208-96-8	530	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
Anthracene	120-12-7	1,800	USEPA Tap Water RSL	10	10	5.0	2.5
Benzo(a)anthracene	56-55-3	0.033 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Benzo(b)fluoranthene	205-99-2	0.034 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.6
Benzo(g,h,i)perylene	191-24-2	120	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
Benzo(k)fluoranthene	207-08-9	0.34 <sup>c</sup>	DTSC Tap Water Screening Level	10	10	5.0	2.5
Benzoic acid	65-85-0	75,000	USEPA Tap Water RSL	100	100	40	20
Benzo(a)pyrene	50-32-8	0.0034 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Benzyl alcohol	100-51-6	2,000	USEPA Tap Water RSL	10	10	5.0	2.5
Benzyl butyl phthalate	85-68-7	16	USEPA Tap Water RSL	10	10	5.0	2.5
bis (2-ethylhexyl)phthalate	117-81-7	4.0 <sup>d</sup>	State of California MCL	10	10	5.0	2.5
bis (2-chloroethoxy) methane	111-91-1	59	USEPA Tap Water RSL	10	10	5.0	2.5
bis (2-chloroethyl) ether	111-44-4	0.014 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
4-Bromophenyl phenyl ether	101-55-3	40	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
Carbazole	86-74-8	310	DTSC Tap Water Screening Level <sup>b</sup>	10	10	5.0	2.5
4-Chloroaniline	106-47-8	0.36 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	4.2
4-Chloro-3-methylphenol	59-50-7	1,400	USEPA Tap Water RSL	10	10	5.0	2.5
2-Chloronaphthalene	91-58-7	750	USEPA Tap Water RSL	10	10	5.0	2.5
2-Chlorophenol	95-57-8	29	DTSC Tap Water Screening Level	10	10	5.0	2.5
4-Chlorophenyl phenylether	7005-72-3	0.36 <sup>e</sup>	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5

## SAP Worksheet #15c—Reference Limits and Evaluation Table

**Matrix: Water**  
**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Chrysene	218-01-9	3.4 <sup>c</sup>	USEPA Tap Water RSL	10	10 <sup>c</sup>	5.0	2.5
Dibenz(a,h)anthracene	53-70-3	0.0034 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Dibenzofuran	132-64-9	7.9 <sup>f</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
1,2-Dichlorobenzene	95-50-1	300	USEPA Tap Water RSL	10	10	5.0	2.5
1,3-Dichlorobenzene	541-73-1	180	DTSC Tap Water Screening Level	10	10	5.0	2.5
1,4-Dichlorobenzene	106-46-7	0.48 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
3,3'-Dichlorobenzidine	91-94-1	0.12 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
2,4-Dichlorophenol	120-83-2	46	USEPA Tap Water RSL	10	10	5.0	2.5
Diethyl phthalate	84-66-2	15,000	USEPA Tap Water RSL	10	10	5.0	2.5
2,4-Dimethylphenol	105-67-9	360	USEPA Tap Water RSL	10	10	5.0	2.6
Dimethyl phthalate	131-11-3	15,000	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
Di-n-butylphthalate	84-74-2	900	USEPA Tap Water RSL	10	10	5.0	2.5
Di-n-octylphthalate	117-84-0	200	USEPA Tap Water RSL	10	10	5.0	2.5
4,6-Dinitro-2-methylphenol	534-52-1	1.5 <sup>e</sup>	USEPA Tap Water RSL	20	20	5.0	2.5
2,4-Dinitrophenol	51-28-5	39	USEPA Tap Water RSL	20	20	5.0	2.5
2,4-Dinitrotoluene	121-14-2	0.24 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
2,6-Dinitrotoluene	606-20-2	0.048 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Fluoranthene	206-44-0	800	USEPA Tap Water RSL	10	10	5.0	2.5
Fluorene	86-73-7	290	USEPA Tap Water RSL	10	10	5.0	2.5
Hexachlorobenzene	118-74-1	0.0098 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Hexachlorobutadiene	87-68-3	0.14 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Hexachloroethane	67-72-1	0.33 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5

## SAP Worksheet #15c—Reference Limits and Evaluation Table

**Matrix: Water**  
**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Indeno(1,2,3-cd)pyrene	193-39-5	0.034 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Isophorone	78-59-1	78	USEPA Tap Water RSL	10	10	5.0	2.5
2-Methylnaphthalene	91-57-6	36	USEPA Tap Water RSL	10	10	5.0	2.5
2-Methylphenol	95-48-7	930	USEPA Tap Water RSL	10	10	5.0	2.5
4-Methylphenol	106-44-5	1,900	USEPA Tap Water RSL	10	10	5.0	2.5
Naphthalene	91-20-3	0.17 <sup>c</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
2-Nitroaniline	88-74-4	190	USEPA Tap Water RSL	10	10	5.0	2.5
3-Nitroaniline	99-09-2	3.8 <sup>d</sup>	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
4-Nitroaniline	100-01-6	3.8 <sup>d</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Nitrobenzene	98-95-3	0.14 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
2-Nitrophenol	88-75-5	39	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
4-Nitrophenol	100-02-7	39	USEPA Tap Water RSL <sup>b</sup>	20	20	5.0	2.5
N-Nitrosodimethylamine	62-75-9	0.00045 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
N-Nitroso-di-n-propylamine	621-64-7	0.011 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
N-Nitrosodiphenylamine	86-30-6	12	USEPA Tap Water RSL	10	10	5.0	2.5
2,2-Oxybis(1-chloropropane)	108-60-1	0.36 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	2.5
Pentachlorophenol	87-86-5	0.04 <sup>e</sup>	USEPA Tap Water RSL	20	20	5.0	2.5
Phenanthrene	85-01-8	1,800	USEPA Tap Water RSL <sup>b</sup>	10	10	5.0	2.5
Phenol	108-95-2	5,800	USEPA Tap Water RSL	10	10	5.0	2.5
Pyrene	129-00-0	120	USEPA Tap Water RSL	10	10	5.0	2.5
1,2,4-Trichlorobenzene	120-82-1	1.1 <sup>e</sup>	USEPA Tap Water RSL	10	10	5.0	3.6

## SAP Worksheet #15c—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
2,4,5-Trichlorophenol	95-95-4	1,200	USEPA Tap Water RSL	10	10	5.0	2.5
2,4,6-Trichlorophenol	88-06-2	1.2 <sup>e</sup>	DTSC Tap Water Screening Level	10	10	5.0	2.5

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal and State of California MCL, the USEPA tap water RSL (USEPA, 2015), and the DTSC-recommended tap water screening levels (DTSC, 2015).

<sup>b</sup> The following surrogate chemicals were used:

- Acenaphthene for acenaphthylene
- Pyrene for benzo(g,h,i)perylene
- bis(2-chloro-1-methylethyl)ether for 4-chloro phenylether
- Pentabromodiphenyl ether for 4-bromo phenyl ether
- 2,4-dinitrophenol for 2-nitrophenol and 4-nitrophenol
- Anthracene for phenanthrene
- Diphenylamine for carbazole
- Diethyl phthalate for dimethyl phthalate
- 4-Nitroaniline for 3-nitroaniline

<sup>c</sup> The LOQ, LOD, or DL do not meet the PAL; however, the analyte will be analyzed by USEPA 8270 SIM.

<sup>d</sup> The LOQ and LOD do not meet the PAL; however, the DL is sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>e</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the PAL, but data evaluation will be based on reported concentrations above the DL.

<sup>f</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL.

## SAP Worksheet #15d—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Acenaphthene	83-32-9	3,600,000	USEPA Residential Soil RSL	333	333	167	83
Acenaphthylene	208-96-8	3,600,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83
Anthracene	120-12-7	18,000,000	USEPA Residential Soil RSL	333	333	167	83
Benzo(a)anthracene	56-55-3	160 <sup>c</sup>	USEPA Residential Soil RSL	333	333	167	83
Benzo(b)fluoranthene	205-99-2	160 <sup>c</sup>	USEPA Residential Soil RSL	333	333	167	86
Benzo(g,h,i)perylene	191-24-2	1,100	USEPA Eco-SSL	333	333	167	87
Benzo(k)fluoranthene	207-08-9	390	DTSC Soil Screening Level	333	333	167	83
Benzoic acid	65-85-0	250,000,000	USEPA Residential Soil RSL	1,333	1,333	667	333
Benzo(a)pyrene	50-32-8	16 <sup>c</sup>	USEPA Residential Soil RSL	333	333	167	83
Benzyl alcohol	100-51-6	6,300,000	USEPA Residential Soil RSL	333	333	167	83
Benzyl butyl phthalate	85-68-7	290,000	USEPA Residential Soil RSL	333	333	167	83
bis (2-ethylhexyl)phthalate	117-81-7	39,000	USEPA Residential Soil RSL	333	333	167	115
bis (2-chloroethoxy) methane	111-91-1	190,000	USEPA Residential Soil RSL	333	333	167	83
bis (2-chloroethyl) ether	111-44-4	230 <sup>d</sup>	USEPA Residential Soil RSL	333	333	167	83
4-Bromophenyl phenyl ether	101-55-3	130,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	90
Carbazole	86-74-8	1,600,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	89
4-Chloroaniline	106-47-8	2,700	USEPA Residential Soil RSL	333	333	167	83
4-Chloro-3-methylphenol	59-50-7	6,300,000	USEPA Residential Soil RSL	333	333	167	83
2-Chloronaphthalene	91-58-7	4,800,000	USEPA Residential Soil RSL	333	333	167	83
2-Chlorophenol	95-57-8	390,000	USEPA Residential Soil RSL	333	333	167	83
4-Chlorophenyl phenylether	7005-72-3	4,900	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83

## SAP Worksheet #15d—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Chrysene	218-01-9	1,100	USEPA Eco-SSL	333	333	167	83
Dibenz(a,h)anthracene	53-70-3	16 <sup>c</sup>	USEPA Residential Soil RSL	333	333	167	83
Dibenzofuran	132-64-9	73,000	USEPA Residential Soil RSL	333	333	167	83
1,2-Dichlorobenzene	95-50-1	1,800,000	USEPA Residential Soil RSL	333	333	167	83
1,3-Dichlorobenzene	541-73-1	240,000	DTSC Soil Screening Level	333	333	167	83
1,4-Dichlorobenzene	106-46-7	2,600	USEPA Residential Soil RSL	333	333	167	83
3,3'-Dichlorobenzidine	91-94-1	1,200	USEPA Residential Soil RSL	333	333	167	84
2,4-Dichlorophenol	120-83-2	190,000	USEPA Residential Soil RSL	333	333	167	83
Diethyl phthalate	84-66-2	51,000,000	USEPA Residential Soil RSL	333	333	167	83
2,4-Dimethylphenol	105-67-9	1,300,000	USEPA Residential Soil RSL	333	333	167	83
Dimethyl phthalate	131-11-3	51,000,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83
Di-n-butylphthalate	84-74-2	6,300,000	USEPA Residential Soil RSL	333	333	167	97
Di-n-octylphthalate	117-84-0	630,000	USEPA Residential Soil RSL	333	333	167	97
4,6-Dinitro-2-methylphenol	534-52-1	5,100	USEPA Residential Soil RSL	667	667	167	83
2,4-Dinitrophenol	51-28-5	130,000	USEPA Residential Soil RSL	667	667	167	86
2,4-Dinitrotoluene	121-14-2	1,700	USEPA Residential Soil RSL	333	333	167	83
2,6-Dinitrotoluene	606-20-2	360	USEPA Residential Soil RSL	333	333	167	83
Fluoranthene	206-44-0	2,400,000	USEPA Residential Soil RSL	333	333	167	126
Fluorene	86-73-7	2,400,000	USEPA Residential Soil RSL	333	333	167	83
Hexachlorobenzene	118-74-1	210 <sup>d</sup>	USEPA Residential Soil RSL	333	333	167	83
Hexachlorobutadiene	87-68-3	1,200	USEPA Residential Soil RSL	333	333	167	83
Hexachloroethane	67-72-1	1,800	USEPA Residential Soil RSL	333	333	167	83

## SAP Worksheet #15d—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Indeno(1,2,3-cd)pyrene	193-39-5	160 <sup>c</sup>	USEPA Residential Soil RSL	333	333	167	83
Isophorone	78-59-1	570,000	USEPA Residential Soil RSL	333	333	167	83
2-Methylnaphthalene	91-57-6	240,000	USEPA Residential Soil RSL	333	333	167	83
2-Methylphenol	95-48-7	3,200,000	USEPA Residential Soil RSL	333	333	167	83
4-Methylphenol	106-44-5	6,300,000	USEPA Residential Soil RSL	333	333	167	83
Naphthalene	91-20-3	3,800	USEPA Residential Soil RSL	333	333	167	83
2-Nitroaniline	88-74-4	630,000	USEPA Residential Soil RSL	333	333	167	83
3-Nitroaniline	99-09-2	27,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83
4-Nitroaniline	100-01-6	27,000	USEPA Residential Soil RSL	333	333	167	120
Nitrobenzene	98-95-3	5,100	USEPA Residential Soil RSL	333	333	167	83
2-Nitrophenol	88-75-5	130,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83
4-Nitrophenol	100-02-7	130,000	USEPA Residential Soil RSL <sup>b</sup>	667	667	167	106
N-Nitrosodimethylamine	62-75-9	2.0 <sup>e</sup>	USEPA Residential Soil RSL	333	333	167	83
N-Nitroso-di-n-propylamine	621-64-7	78 <sup>e</sup>	USEPA Residential Soil RSL	333	333	167	83
N-Nitrosodiphenylamine	86-30-6	110,000	USEPA Residential Soil RSL	333	333	167	153
2,2-Oxybis(1-chloropropane)	108-60-1	4,900	USEPA Residential Soil RSL	333	333	167	83
Pentachlorophenol	87-86-5	1,000	USEPA Residential Soil RSL	667	667	167	83
Phenanthrene	85-01-8	18,000,000	USEPA Residential Soil RSL <sup>b</sup>	333	333	167	83
Phenol	108-95-2	19,000,000	USEPA Residential Soil RSL	333	333	167	83
Pyrene	129-00-0	1,800,000	USEPA Residential Soil RSL	333	333	167	160
1,2,4-Trichlorobenzene	120-82-1	24,000	USEPA Residential Soil RSL	333	333	167	83

## SAP Worksheet #15d—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: SVOCs – USEPA Method 8270C**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
2,4,5-Trichlorophenol	95-95-4	6,300,000	USEPA Residential Soil RSL	333	333	167	91
2,4,6-Trichlorophenol	88-06-2	57,500	USEPA Residential Soil RSL	333	333	167	83

Notes:

<sup>a</sup> The PAL is the most conservative value from among the USEPA residential soil RSL (USEPA, 2015), USEPA Eco-SSLs (USEPA, 2008), and the DTSC soil screening levels (DTSC, 2015).

<sup>b</sup> The following surrogate chemicals were used:

- Acenaphthene for acenaphthylene
- Pentabromodiphenyl ether for 4-bromo phenyl ether
- bis(2-chloro-1-methylethyl)ether for 4-chlorophenyl phenylether
- 2,4-dinitrophenol for 2-nitrophenol and 4-nitrophenol
- Anthracene for phenanthrene
- Diethyl phthalate for dimethyl phthalate
- Diphenylamine for carbazole
- 4-Nitroaniline for 3-nitroaniline

<sup>c</sup> The LOQ, LOD, or DL do not meet the PAL; however, the analyte will be analyzed by USEPA 8270SIM.

<sup>d</sup> The LOQ does not meet the PAL; however, the DL is sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>e</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the USEPA Res Soil RSL, but data evaluation will be based on reported concentrations above the DL.

Eco-SSL = ecological soil screening level

## SAP Worksheet #15e—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: PAHs – USEPA Method 8270C SIM**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Naphthalene	91-20-3	0.17 <sup>b</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Acenaphthene	83-32-9	530	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Acenaphthylene	208-96-8	530	USEPA Tap Water RSL <sup>c</sup>	0.5	0.5	0.1	0.05
Fluorene	86-73-7	290	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Phenanthrene	85-01-8	1,800	USEPA Tap Water RSL <sup>c</sup>	0.5	0.5	0.1	0.05
Anthracene	120-12-7	1,800	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Fluoranthene	206-44-0	800	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Pyrene	129-00-0	120	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Benzo(a)anthracene	56-55-3	0.033 <sup>d</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.09
Chrysene	218-01-9	3.4	DTSC Tap Water Screening Level	0.5	0.5	0.1	0.06
Benzo(b)fluoranthene	205-99-2	0.034 <sup>d</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Benzo(k)fluoranthene	207-08-9	0.34 <sup>b</sup>	DTSC Tap Water Screening Level	0.5	0.5	0.1	0.05
Benzo(a)pyrene	50-32-8	0.0034 <sup>d</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Indeno(1,2,3-cd)pyrene	193-39-5	0.034 <sup>d</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Dibenzo(a,h)anthracene	53-70-3	0.0034 <sup>d</sup>	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
Benzo(g,h,i)perylene	191-24-2	120	USEPA Tap Water RSL <sup>c</sup>	0.5	0.5	0.1	0.05

## SAP Worksheet #15e—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: PAHs – USEPA Method 8270C SIM**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1-Methylnaphthalene	90-12-0	1.1	USEPA Tap Water RSL	0.5	0.5	0.1	0.05
2-Methylnaphthalene	91-57-6	36	USEPA Tap Water RSL	0.5	0.5	0.1	0.05

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal and State of California MCL, the USEPA tap water RSL (USEPA, 2015), and the DTSC-recommended tap water level (DTSC, 2015).

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>c</sup> The following surrogate chemicals were used:

Anthracene for phenanthrene

Acenaphthene for acenaphthylene

Pyrene for benzo(g,h,i)perylene

<sup>d</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but will be qualified as an estimated value (“J” flagged). There is a level of uncertainty between the DL and the PAL, but data evaluation will be based on reported concentrations above the DL.

<sup>e</sup> The LOQ and LOD do not meet the PAL; however, the DL is sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

## SAP Worksheet #15f—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: PAHs – USEPA Method 8270C SIM**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Naphthalene	91-20-3	3,800	USEPA Residential Soil RSL	10	10	2.5	1.25
Acenaphthene	83-32-9	3,600,000	USEPA Residential Soil RSL	10	10	2.5	1.25
Acenaphthylene	208-96-8	3,600,000	USEPA Residential Soil RSL <sup>b</sup>	10	10	2.5	1.25
Fluorene	86-73-7	2,400,000	USEPA Residential Soil RSL	10	10	2.5	1.25
Phenanthrene	85-01-8	18,000,000	USEPA Residential Soil RSL <sup>b</sup>	10	10	2.5	1.25
Anthracene	120-12-7	18,000,000	USEPA Residential Soil RSL	10	10	2.5	1.25
Fluoranthene	206-44-0	2,400,000	USEPA Residential Soil RSL	10	10	2.5	1.25
Pyrene	129-00-0	1,800,000	USEPA Residential Soil RSL	10	10	2.5	1.25
Benzo(a)anthracene	56-55-3	160	USEPA Residential Soil RSL	10	10	2.5	2.45
Chrysene	218-01-9	1,100	USEPA Eco-SSL	10	10	2.5	2.20
Benzo(b)fluoranthene	205-99-2	160	USEPA Residential Soil RSL	10	10	2.5	1.25
Benzo(k)fluoranthene	207-08-9	390	DTSC Soil Screening Level	10	10	2.5	1.25
Benzo(a)pyrene	50-32-8	16	USEPA Residential Soil RSL	10	10	2.5	1.25
Indeno(1,2,3-cd)pyrene	193-39-5	160	USEPA Residential Soil RSL	10	10	2.5	1.25
Dibenzo(a,h)anthracene	53-70-3	16	USEPA Residential Soil RSL	10	10	2.5	1.25
Benzo(g,h,i)perylene	191-24-2	1,100	USEPA Eco-SSL	10	10	2.5	1.25
1-Methylnaphthalene	90-12-0	18,000	USEPA Residential Soil RSL	10	10	2.5	1.25
2-Methylnaphthalene	91-57-6	240,000	USEPA Residential Soil RSL	10	10	2.5	1.25

Notes:

<sup>a</sup> The PAL is the most conservative value from among the USEPA residential soil RSL (USEPA, 2015), USEPA Eco-SSLs (USEPA, 2008), and the DTSC soil screening level (DTSC, 2015).

<sup>b</sup> The following surrogate chemicals were used:

- Acenaphthene for acenaphthylene
- Anthracene for phenanthrene

## SAP Worksheet #15g—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: TPH – USEPA Method 8015B**

Analyte	CAS Number	Project Action Limit (mg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (mg/L)	Laboratory-Specific Limits		
					LOQ (mg/L)	LOD (mg/L)	DL (mg/L)
TPH-e as diesel	68476-34-6	0.1 <sup>b</sup>	SFBRWQCB ESL	0.5	0.5	0.05	0.025
TPH-e as motor oil	8002-05-9	0.1 <sup>b</sup>	SFBRWQCB ESL	0.5	0.5	0.05	0.025
TPH-p as gasoline	8006-61-9	0.1	SFBRWQCB ESL	0.1	0.1	0.01	0.005

Notes:

<sup>a</sup> The PAL is the SFRWQCB ESL for groundwater (SFBRWQCB, 2013) that is a current or potential drinking water resource.

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated ("J" flagged) between LOQ and DL.

ESL = environmental screening level

mg/L = milligram per liter

SFBRWQCB = San Francisco Bay Regional Water Quality Control Board

## SAP Worksheet #15h—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: TPH – USEPA Method 8015B**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (mg/kg)	Laboratory-Specific Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TPH-e as diesel	68476-34-6	100	SFBRWQCB ESL	10	10	5.0	2.5
TPH-e as motor oil	8002-05-9	100	SFBRWQCB ESL	20	20	5.0	2.5
TPH-p as gasoline	8006-61-9	100	SFBRWQCB ESL	1.0	1.0	0.5	0.25

Note:

<sup>a</sup> The PAL is the SFBRWQCB ESL for residential exposure to shallow soil (SFBRWQCB, 2013) where groundwater is a current or potential drinking water resource.

## SAP Worksheet #15i—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: OCPs – USEPA Method 8081A**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
4,4'-DDD	72-54-8	0.031 <sup>b</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
4,4'-DDE	72-55-9	0.046 <sup>b</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
4,4'-DDT	50-29-3	0.23	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
Aldrin	309-00-2	0.00092 <sup>c</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
alpha- BHC	319-84-6	0.0071 <sup>d</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
alpha-Chlordane	5103-71-9	0.045 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	0.1	0.1	0.01	0.005
beta-BHC	319-85-7	0.025 <sup>b</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.007
delta-BHC	319-86-8	0.025 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	0.1	0.1	0.01	0.007
Dieldrin	60-57-1	0.0017 <sup>c</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
Endosulfan I	959-98-8	100	USEPA Tap Water RSL	0.1	0.1	0.01	0.008
Endosulfan II	33213-65-9	100	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
Endosulfan sulfate	1031-07-8	100	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
Endrin	72-20-8	2.0	State and Federal MCL	0.1	0.1	0.01	0.008
Endrin aldehyde	7421-93-4	2.0	State and Federal MCL	0.1	0.1	0.01	0.005
Endrin ketone	53494-70-5	2.0	State and Federal MCL	0.1	0.1	0.01	0.005
gamma-BHC (Lindane)	58-89-9	0.041 <sup>b</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005
gamma-Chlordane	5566-34-7	0.045 <sup>b</sup>	USEPA Tap Water RSL <sup>e</sup>	0.1	0.1	0.01	0.005
Heptachlor	76-44-8	0.0014 <sup>c</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.007
Heptachlor epoxide	1024-57-3	0.0014 <sup>c</sup>	USEPA Tap Water RSL	0.1	0.1	0.01	0.005

## SAP Worksheet #15i—Reference Limits and Evaluation Table

### Matrix: Water

### Analytical Group: OCPs – USEPA Method 8081A

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Methoxychlor	72-43-5	30	State of California MCL	1.0	1.0	0.1	0.050
Toxaphene	8001-35-2	0.015 <sup>c</sup>	USEPA Tap Water RSL	2.0	2.0	0.5	0.250
Technical Chlordane	12789-03-6	0.045 <sup>c</sup>	USEPA Tap Water RSL <sup>e</sup>	1.0	1.0	0.25	0.125

#### Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal and State of California MCL (CDPH, 2014), the USEPA tap water RSL (USEPA, 2015), and the DTSC-recommended tap water levels (DTSC, 2015).

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>c</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the USEPA tap water RSL, but data evaluation will be based on reported concentrations above the DL.

<sup>d</sup> The LOQ and LOD do not meet the PAL; however, the DL is sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>e</sup> The following surrogate chemicals were used:

- beta-BHC for delta-BHC
- Chlordane for alpha-chlordane, gamma-chlordane, and technical chlordane
- Endosulfan for endosulfan I, endosulfan II, and endosulfan sulfate
- Endrin for endrin aldehyde and endrin ketone

BHC = benzene hexachloride

DDD = dichlorodiphenyldichloroethane

DDE = dichlorodiphenyldichloroethene

DDT = dichlorodiphenyltrichloroethane

## SAP Worksheet #15j—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: OCPs – USEPA Method 8081A**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
4,4'-DDD	72-54-8	2,300	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
4,4'-DDE	72-55-9	2,000	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
4,4'-DDT	50-29-3	1,900	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
Aldrin	309-00-2	39	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
alpha- BHC	319-84-6	86	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
alpha-Chlordane	5103-71-9	430	DTSC Soil Screening Level <sup>b</sup>	2.0	2.0	0.4	0.2
beta-BHC	319-85-7	300	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
delta-BHC	319-86-8	300	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.27
Dieldrin	60-57-1	34	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
Endosulfan I	959-98-8	470,000	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.2
Endosulfan II	33213-65-9	470,000	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.2
Endosulfan sulfate	1031-07-8	470,000	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.2
Endrin	72-20-8	19,000	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
Endrin aldehyde	7421-93-4	19,000	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.35
Endrin ketone	53494-70-5	19,000	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.2
gamma-BHC (Lindane)	58-89-9	570	USEPA Residential Soil RSL <sup>b</sup>	2.0	2.0	0.4	0.2
gamma-Chlordane	5566-34-7	430	DTSC Soil Screening Level <sup>b</sup>	2.0	2.0	0.4	0.2
Heptachlor	76-44-8	130	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2
Heptachlor epoxide	1024-57-3	70	USEPA Residential Soil RSL	2.0	2.0	0.4	0.2

## SAP Worksheet #15j—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: OCPs – USEPA Method 8081A**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Methoxychlor	72-43-5	320,000	USEPA Residential Soil RSL	10	10	4.0	2.0
Toxaphene	8001-35-2	490	USEPA Residential Soil RSL	50	50	10	5.0
Technical Chlordane	12789-03-6	430	DTSC Soil Screening Level <sup>b</sup>	50	50	20	10

Notes:

<sup>a</sup> The PAL is the most conservative value from among the USEPA residential soil RSL (USEPA, 2015), USEPA Eco-SSLs (USEPA, 2008), and the DTSC soil screening levels (DTSC, 2015).

<sup>b</sup> The following surrogate chemicals were used:

- beta-BHC for delta-BHC
- Chlordane for alpha-chlordane, gamma-chlordane, and technical chlordane
- Endosulfan for endosulfan I, endosulfan II, and endosulfan sulfate
- Endrin for endrin aldehyde and endrin ketone

## SAP Worksheet #15k—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: PCBs – USEPA Method 8082**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aroclor 1016	12674-11-2	0.22 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.45
Aroclor 1221	11104-28-2	0.0046 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.29
Aroclor 1232	11141-16-5	0.0046 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Aroclor 1242	53469-21-9	0.0078 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Aroclor 1248	12672-29-6	0.0078 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Aroclor 1254	11097-69-1	0.0078 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Aroclor 1260	11096-82-5	0.0078 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.5	0.31

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal (USEPA, 2015) and State of California MCL (CDPH, 2014), the USEPA tap water RSL (USEPA, 2015), and DTSC-recommended tap water screening levels (DTSC, 2015).

<sup>b</sup> The LOQ, LOD, and DL do not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory's DoD ELAP-accredited methods. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated ("J" flagged). There is a level of uncertainty between the DL and the USEPA tap water RSL, but data evaluation will be based on reported concentrations above the DL.

## SAP Worksheet #15I—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: PCBs – USEPA Method 8082**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Aroclor 1016	12674-11-2	230	DTSC Soil Screening Level	50	50	16.7	13
Aroclor 1221	11104-28-2	170	USEPA Residential Soil RSL	50	50	16.7	8.3
Aroclor 1232	11141-16-5	170	USEPA Residential Soil RSL	50	50	16.7	9.0
Aroclor 1242	53469-21-9	230	USEPA Residential Soil RSL	50	50	16.7	9.3
Aroclor 1248	12672-29-6	230	USEPA Residential Soil RSL	50	50	16.7	8.3
Aroclor 1254	11097-69-1	20	USEPA Residential Soil RSL	50	50	16.7	8.3
Aroclor 1260	11096-82-5	240	USEPA Residential Soil RSL	50	50	16.7	9.9

Note:

<sup>a</sup> The PAL is the most conservative value between the USEPA residential soil RSL (USEPA, 2015) and the DTSC soil screening levels (DTSC, 2015).

## SAP Worksheet #15m—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: Total and Dissolved Metals – USEPA Methods 6020A and 7470A**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aluminum	7429-90-5	1,000	State of California MCL	100	100	20	10
Antimony	7440-36-0	6.0	State of California MCL	1.0	1.0	0.5	0.25
Arsenic	7440-38-2	0.052 <sup>b</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Barium	7440-39-3	1,000	State of California MCL	1.0	1.0	0.5	0.25
Beryllium	7440-41-7	2.5	USEPA Tap Water RSL	1.0	1.0	0.1	0.05
Cadmium	7440-43-9	5.0	State and Federal MCL	1.0	1.0	0.2	0.1
Calcium	7440-70-2	NA	NA	100	100	25	13
Chromium	7440-47-3	50	State of California MCL <sup>c</sup>	1.0	1.0	0.2	0.1
Cobalt	7440-48-4	6.0	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Copper	7440-50-8	800	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Iron	7439-89-6	14,000	USEPA Tap Water RSL	100	100	10	5.0
Lead	7439-92-1	15	State and Federal MCL	1.0	1.0	0.1	0.05
Magnesium	7439-95-4	NA	NA	100	100	10	5.0
Manganese	7439-96-5	430	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Mercury	7439-97-6	2.0	DTSC Tap Water Screening Level <sup>c</sup>	0.5	0.5	0.1	0.05
Molybdenum	7439-98-7	100	USEPA Tap Water RSL	2.0	2.0	0.5	0.25
Nickel	7440-02-0	100	State of California MCL	1.0	1.0	0.2	0.1
Potassium	7440-09-7	NA	NA	100	100	20	10
Selenium	7782-49-2	50	State and Federal MCL	1.0	1.0	0.3	0.15
Silver	7440-22-4	94	USEPA Tap Water RSL	1.0	1.0	0.2	0.1

## SAP Worksheet #15m—Reference Limits and Evaluation Table

### Matrix: Water

### Analytical Group: Total and Dissolved Metals – USEPA Methods 6020A and 7470A

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Sodium	7440-23-5	NA	NA	100	100	50	25
Thallium	7440-28-0	0.2 <sup>d</sup>	USEPA Tap Water RSL	1.0	1.0	0.2	0.1
Vanadium	7440-62-2	86	USEPA Tap Water RSL	1.0	1.0	0.5	0.25
Zinc	7440-66-6	6,000	USEPA Tap Water RSL	20	20	10	5.0

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal (USEPA, 2015) and State of California MCL (CDPH, 2014), the USEPA tap water RSL (USEPA, 2015), and the DTSC-recommended tap water screening levels (DTSC, 2015).

<sup>b</sup> The LOQ, LOD, and DL do not meet the PAL for arsenic; however, arsenic occurs naturally at concentrations that exceed the tap water RSL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the PAL, but data evaluation will be based on reported concentrations above the DL.

<sup>c</sup> The MCL for total chromium is used for chromium. Methyl mercury was used for the mercury PAL.

<sup>d</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

NA = not applicable

## SAP Worksheet #15n—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Total Metals – USEPA Methods 6020A and 7471A**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (mg/kg)	Laboratory-Specific Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aluminum	7429-90-5	77,000	USEPA Residential Soil RSL	100	100	10	5.0
Antimony	7440-36-0	0.27 <sup>b</sup>	USEPA Eco-SSL	0.5	0.5	0.2	0.1
Arsenic	7440-38-2	0.11 <sup>c</sup>	DTSC Soil Screening Level	0.5	0.5	0.1	0.05
Barium	7440-39-3	330	USEPA Eco-SSL	0.5	0.5	0.1	0.072
Beryllium	7440-41-7	3.0	DTSC Soil Screening Level	0.5	0.5	0.1	0.05
Cadmium	7440-43-9	0.36 <sup>b</sup>	USEPA Eco-SSL	0.5	0.5	0.1	0.057
Calcium	7440-70-2	NA	NA	100	100	20	17
Chromium	7440-47-3	26	USEPA Eco-SSL	0.5	0.5	0.1	0.05
Cobalt	7440-48-4	13	USEPA Eco-SSL	0.5	0.5	0.1	0.05
Copper	7440-50-8	28	USEPA Eco-SSL	0.5	0.5	0.2	0.1
Iron	7439-89-6	55,000	USEPA Residential Soil RSL	100	100	10	5.0
Lead	7439-92-1	11	USEPA Eco-SSL	0.5	0.5	0.1	0.05
Magnesium	7439-95-4	NA	NA	100	100	20	10
Manganese	7439-96-5	220	USEPA Eco-SSL	0.5	0.5	0.2	0.15
Mercury	7439-97-6	7.8	USEPA Residential Soil RSL <sup>d</sup>	0.1	0.1	0.02	0.01
Molybdenum	7439-98-7	390	USEPA Residential Soil RSL	0.5	0.5	0.2	0.1
Nickel	7440-02-0	0.42 <sup>b</sup>	DTSC Soil Screening Level	0.5	0.5	0.1	0.063
Potassium	7440-09-7	NA	NA	100	100	20	10
Selenium	7782-49-2	0.52	USEPA Eco-SSL	0.5	0.5	0.1	0.05
Silver	7440-22-4	4.2	USEPA Eco-SSL	0.5	0.5	0.1	0.05
Sodium	7440-23-5	NA	NA	100	100	20	10

## SAP Worksheet #15n—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Total Metals – USEPA Methods 6020A and 7471A**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (mg/kg)	Laboratory-Specific Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Thallium	7440-28-0	0.78	USEPA Residential Soil RSL	0.5	0.5	0.1	0.05
Vanadium	7440-62-2	7.8	USEPA Eco-SSL	0.5	0.5	0.25	0.19
Zinc	7440-66-6	46	USEPA Eco-SSL	2.0	2.0	1.0	0.683

Notes:

<sup>a</sup> The PAL is the most conservative value from among the USEPA residential soil RSL (USEPA, 2015), USEPA Eco-SSLs (USEPA, 2008), and the DTSC soil screening levels (DTSC, 2015).

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged) between LOQ and DL.

<sup>c</sup> The LOQ and LOD do not meet the PAL for arsenic; however, arsenic occurs naturally at concentrations that exceed the tap water RSL. The laboratory will report to the lowest reporting limit (DL), but the value will be qualified as estimated (“J” flagged). There is a level of uncertainty between the DL and the PAL, but data evaluation will be based on reported concentrations above the DL.

<sup>d</sup> Methyl mercury was used for the mercury PAL.

## SAP Worksheet #15o—Reference Limits and Evaluation Table

**Matrix: Water**

**Analytical Group: Hexavalent Chromium – USEPA Method 218.6**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/L)	Laboratory-Specific Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Hexavalent Chromium	18540-29-9	0.035 <sup>b</sup>	USEPA Tap Water RSL	0.05	0.05	0.025	0.017

Notes:

<sup>a</sup> The PAL is the most conservative value from among the federal (USEPA, 2015) and State of California MCL (CDPH, 2014), the USEPA tap water RSL (USEPA, 2015), and the DTSC-recommended tap water values (DTSC, 2015).

<sup>b</sup> The LOQ does not meet the PAL; however, the LOD and DL are sufficient to meet the PAL.

## SAP Worksheet #15p—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Hexavalent Chromium – USEPA Method 7199**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Hexavalent Chromium	18540-29-9	300	USEPA Residential Soil RSL	100	100	40	13

Note:

<sup>a</sup> The PAL is the most conservative value from among the USEPA residential soil RSL (USEPA, 2015), USEPA Eco-SSLs (USEPA, 2008), and the DTSC soil screening levels (DTSC, 2015).

## SAP Worksheet #15q—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Dioxins/Furans – USEPA Method 8290A**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD <sup>b</sup> (µg/kg)	DL <sup>b</sup> (µg/kg)
2,3,7,8-TCDD	1746-01-6	0.0048 <sup>c</sup>	USEPA Residential Soil RSL	0.005	0.005	NA	NA
1,2,3,7,8-PeCDD	40321-76-4	NA	NA	0.0125	0.0125	NA	NA
1,2,3,4,7,8-HxCDD	39227-28-6	NA	NA	0.0125	0.0125	NA	NA
1,2,3,6,7,8-HxCDD	57653-85-7	NA	NA	0.0125	0.0125	NA	NA
1,2,3,7,8,9-HxCDD	19408-74-3	NA	NA	0.0125	0.0125	NA	NA
1,2,3,4,6,7,8-HpCDD	35822-39-4	NA	NA	0.0125	0.0125	NA	NA
OCDD	3268-87-9	NA	NA	0.025	0.025	NA	NA
2,3,7,8-TCDF	51207-31-9	NA	NA	0.005	0.005	NA	NA
1,2,3,7,8-PeCDF	57117-41-6	NA	NA	0.0125	0.0125	NA	NA
2,3,4,7,8-PeCDF	57117-31-4	NA	NA	0.0125	0.0125	NA	NA
1,2,3,4,7,8-HxCDF	70648-26-9	NA	NA	0.0125	0.0125	NA	NA
1,2,3,6,7,8-HxCDF	57117-44-9	NA	NA	0.0125	0.0125	NA	NA
1,2,3,7,8,9-HxCDF	72918-21-9	NA	NA	0.0125	0.0125	NA	NA
2,3,4,6,7,8-HxCDF	60851-34-5	NA	NA	0.0125	0.0125	NA	NA
1,2,3,4,6,7,8-HpCDF	67562-39-4	NA	NA	0.0125	0.0125	NA	NA
1,2,3,4,7,8,9-HpCDF	55673-89-7	NA	NA	0.0125	0.0125	NA	NA
OCDF	39001-02-0	NA	NA	0.025	0.025	NA	NA
Total TCDDs	NL	NA	NA	0.005	0.005	NA	NA
Total PeCDDs	NL	NA	NA	0.0125	0.0125	NA	NA
Total HxCDDs	NL	NA	NA	0.0125	0.0125	NA	NA
Total HpCDDs	NL	NA	NA	0.0125	0.0125	NA	NA

## SAP Worksheet #15q—Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Dioxins/Furans – USEPA Method 8290A**

Analyte	CAS Number	Project Action Limit (µg/kg)	Project Action Limit Reference <sup>a</sup>	Project QL Goal (µg/kg)	Laboratory-Specific Limits		
					LOQ (µg/kg)	LOD <sup>b</sup> (µg/kg)	DL <sup>b</sup> (µg/kg)
Total TCDFs	NL	NA	NA	0.005	0.005	NA	NA
Total PeCDFs	NL	NA	NA	0.0125	0.0125	NA	NA
Total HxCDFs	NL	NA	NA	0.0125	0.0125	NA	NA
Total HpCDFs	NL	NA	NA	0.0125	0.0125	NA	NA

Notes:

<sup>a</sup> The value shown is the USEPA residential soil RSL (USEPA, 2015).

<sup>b</sup> LOD/DL is not applicable to dioxin/furan analytes reported via USEPA Method 8290A. Dioxin/furan results will be reported using the EDL and EMPC limits per the method, as follows:

- For analytes that do not have a chromatographic response in the samples, the results will be reported to the EDL with a “U” laboratory qualifier.
- For dioxin/furan analytes that do have a chromatographic response but that do not meet the method requirements for sample identification, the results will be reported to the EMPC with a “U” laboratory qualifier.
- For dioxin/furan analytes that are positively identified per the method requirements, the results will be reported to the EMPC.
- Quarterly LOD checks are performed to maintain compliance with the DoD QSM Version 5.0 (2013).

<sup>c</sup> The LOQ does not meet the PAL; however, the limits will be the lowest achievable using the best available technology by the laboratory’s DoD ELAP-accredited methods.

EDL = estimated detection limit

EMPC = estimated maximum potential concentration

HpCDD = heptachlorodibenzo-p-dioxin

HpCDF = heptachlorodibenzofuran

HxCDD = hexachlorodibenzo-p-dioxin

HxCDF = hexachlorodibenzofuran

OCDD = 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin

OCDF = octachlorodibenzofuran

PeCDD = pentachlorodibenzo-p-dioxin

PeCDF = pentachlorodibenzofuran

QSM = *Quality Systems Manual for Environmental Laboratories*

TCDD = tetrachlorodibenzo-p-dioxin

TCDF = tetrachlorodibenzofuran

### SAP Worksheet #16—Project Schedule/Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date of Initiation	Anticipated Date of Completion		
Review of work plan with SAP	NAVFAC Southwest, DTSC, RWQCB	July 31, 2014	October 12, 2015	Final work plan with SAP	October 12, 2015
Field sampling	KCH	October 16, 2015	December 1, 2015	Field documentation (field daily reports, field logs and forms)	December 1, 2015
Analytical laboratory	EMAX, APPL	November 6, 2015	December 19, 2015	Data reports (hard-copy and EDD) by SDG	December 19, 2015
Analytical data verification and validation	LDC	December 21, 2015	January 13, 2016	100 percent reviewed analytical data validation reports (hard-copy and validated EDD)	January 13, 2016
RI Report	NAVFAC Southwest, DTSC	January 13, 2016	March 18, 2016	Pre-Draft Report	March 18, 2016

Note:  
 SDG = sample delivery group

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## SAP Worksheet #17—Sampling Design and Rationale

The sampling design and rationale were specifically developed to address the objectives of this investigation. The overall objectives of this investigation are to assess whether the nature and extent of debris and COPCs have been adequately characterized at IRP Site 6 and whether COPCs pose an unacceptable risk to human health or the environment. To meet this objective, the goals of this investigation are to answer the following questions:

- Is the nature and extent of debris at IRP Site 6 adequately characterized?
- Is IRP Site 6 adequately delineated?
- Do concentrations of chemicals in soil or groundwater pose an unacceptable risk to human health or the environment?
- Is there evidence of VOCs, TPH, or dioxins/furans in soil in the trenches?

Table 17-1 presents the rationale used to select soil boring locations. Table 17-2 presents the rationale used to select monitoring well locations. Figure 10-3 shows the proposed sampling locations. The sampling and analytical program consists of 12 soil borings and 12 trenches that will be installed and sampled as follows:

- Five borings (KCH06-HA01 to KCH06-HA05) installed via hand auger to an estimated depth of 10 feet bgs, each with one surface soil sample collected between ground surface and 0.5 foot bgs, and two subsurface soil samples collected between 4 and 6 feet bgs and 8 and 10 feet bgs.
- Five borings (KCH06-SB01 to KCH06-SB05) installed by HSA to an estimated depth of 20 feet bgs, each with one surface soil sample collected between ground surface and 0.5 foot bgs, and two subsurface soil samples collected between 4 and 6 feet bgs and 8 and 10 feet bgs. Two additional subsurface soil samples will be collected at approximately 14 to 16 feet bgs and 18 to 20 feet bgs.
- Twelve trenches (KCH06-TR01 to KCH06-TR12) will be excavated to an approximate depth of 10 feet bgs, with an average of three soil samples collected per trench. Sample collection depth will depend on site conditions; however, surface (0 to 0.5 foot bgs) and subsurface samples will be collected.
- Two monitoring wells (KCH06-MW01 and KCH06-MW02) will be installed to an estimated depth of 150 feet bgs. During well installation, up to 12 soil samples will be collected from each boring. Soil samples will be collected from 0 to 0.5 foot bgs, 4 to 6 feet bgs, and 8 to 10 feet bgs. Additional soil samples will be collected at 5-foot intervals from 10 feet bgs to 30 feet bgs and at 20-foot intervals to groundwater.
- The two new monitoring wells (KCH06-MW01 and KCH06-MW02) and two existing monitoring wells (SB-10 and B-21) will be sampled during two events. The new monitoring wells will be developed prior to sampling.

Soil and groundwater sample analyses will include the following:

- Soil - SVOCs by USEPA 8270C, PAHs by USEPA 8270C SIM, OCPs by USEPA 8081A, PCBs by USEPA 8082, metals by USEPA 6020A/7471A, and hexavalent chromium by

USEPA 7199, VOCs by USEPA 8260B, TPH-p and TPH-e by USEPA 8015B, and dioxins/furans by USEPA 8290A are contingency analyses. Soil samples may be analyzed for these methods if there is visible contamination, odors, or PID readings in excess of 5 ppm, or visible evidence of prior burning (dioxins/furans).

- Groundwater - VOCs by USEPA 8260B, SVOCs by USEPA 8270C, PAHs by USEPA 8270C SIM, TPH-p and TPH-e by USEPA 8015B, OCPs by USEPA 8081A, PCBs by USEPA 8082, total and dissolved metals by USEPA 6020A/7470A, and hexavalent chromium by USEPA 218.6.

**Table 17-1. Rationale of Proposed Investigation Locations – Soil**

Location ID	Rationale
KCH06-HA01	Hand auger soil boring located near the downstream (south) end of South Ravine to evaluate site impacts on soil.
KCH06-HA02	Hand auger soil boring located in the southern portion of the site, upstream (north) from KCH06-HA01 to evaluate site impacts on soil.
KCH06-HA03	Hand auger soil boring located near the downstream end of the northeast branch to evaluate site impacts on soil.
KCH06-HA04	Hand auger soil boring located in the northeast branch of the ravine, upstream from KCH06-HA03 to evaluate site impacts on soil.
KCH06-HA05	Hand auger soil boring located near the upstream end of the northeast branch of the ravine to evaluate site impacts on soil.
KCH06-SB01	Soil boring located in the southern portion of the site, upstream from KCH06-TR01 to evaluate site impacts on soil.
KCH06-SB02	Soil boring located near the upstream end of the northwest branch of the ravine to evaluate site impacts on soil.
KCH06-SB03	Soil boring located between the northwest and northeast branches of the ravine to evaluate site impacts on soil.
KCH06-SB04	Soil boring located in the northeast branch of the ravine to evaluate site impacts on soil.
KCH06-SB05	Soil boring located between the northwest and northeast branches of the ravine, upstream from KCH06-SB03 to evaluate site impacts on soil.
KCH06-TR01	Trench located in the southern portion of the site, upstream (north) from KCH06-HA02 to evaluate the extent of debris and site impacts on soil.
KCH06-TR02	Trench located in the southern portion of the site to evaluate the extent of debris and site impacts on soil.
KCH06-TR03	Trench located near the downstream end of the northwest branch to evaluate the extent of debris and site impacts on soil.
KCH06-TR04	Trench located southwest of the northwest branch of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR05	Trench located in the northwest branch of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR06	Trench located in the northwest branch of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR07	Trench located between the northwest and northeast branches of the ravine to evaluate the extent of debris and site impacts on soil.

**Table 17-1. Rationale of Proposed Investigation Locations – Soil**

KCH06-TR08	Trench located near the upstream end of the northeast branch of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR09	Trench located between the northwest and northeast branches of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR10	Trench located between the northwest and northeast branches of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR11	Trench located near the upstream end of the northeast branch of the ravine to evaluate the extent of debris and site impacts on soil.
KCH06-TR12	Trench located east of the northeast branch of the ravine to evaluate the extent of debris and site impacts on soil.

**Table 17-2. Rationale of Proposed Investigation Locations – Groundwater**

Location ID	Rationale
SB-10	Existing monitoring well located upstream and northwest of IRP Site 6 to evaluate site impacts on groundwater.
B-21	Existing monitoring well located at the southern boundary of IRP Site 6 to evaluate site impacts on groundwater.
KCH06-MW01	New monitoring well located near the northwest branch of the ravine to evaluate site impacts on soil and groundwater.
KCH06-MW02	New monitoring well located near the intersection of the northwest and northeast branches of the ravine to evaluate site impacts on soil and groundwater.

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### SAP Worksheet #18—Location-Specific Sampling Methods/SOP Requirements Table

Sampling Location/ ID Number <sup>a</sup>	Matrix	Depth <sup>b</sup> (feet bgs)	Analytical Group	Sampling Frequency	Number of Samples (Identify Field Duplicates) <sup>d</sup>	Sampling SOP Reference
KCH06-HA01	Soil	0 to 0.5 4 to 6 8 to 10	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Three per boring	See Worksheet #14
KCH06-HA02	Soil	0 to 0.5 4 to 6 8 to 10	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Three per boring	See Worksheet #14
KCH06-HA03	Soil	0 to 0.5 4 to 6 8 to 10	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Three per boring	See Worksheet #14
KCH06-HA04	Soil	0 to 0.5 4 to 6 8 to 10	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Three per boring	See Worksheet #14
KCH06-HA05	Soil	0 to 0.5 4 to 6 8 to 10	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Three per boring	See Worksheet #14
KCH06-MW01	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20 23 to 25 28 to 30 48 to 50 68 to 70 88 to 90 98 to 110 118 to 120	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to 12 per boring	See Worksheet #14

### SAP Worksheet #18—Location-Specific Sampling Methods/SOP Requirements Table

Sampling Location/ ID Number <sup>a</sup>	Matrix	Depth <sup>b</sup> (feet bgs)	Analytical Group	Sampling Frequency	Number of Samples (Identify Field Duplicates) <sup>d</sup>	Sampling SOP Reference
KCH06-MW02	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20 23 to 25 28 to 30 48 to 50 68 to 70 88 to 90 98 to 110 118 to 120	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to 12 per boring	See Worksheet #14
KCH06-SB01	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to five per boring	See Worksheet #14
KCH06-SB02	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to five per boring	See Worksheet #14
KCH06-SB03	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to five per boring	See Worksheet #14

### SAP Worksheet #18—Location-Specific Sampling Methods/SOP Requirements Table

Sampling Location/ ID Number <sup>a</sup>	Matrix	Depth <sup>b</sup> (feet bgs)	Analytical Group	Sampling Frequency	Number of Samples (Identify Field Duplicates) <sup>d</sup>	Sampling SOP Reference
KCH06-SB04	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to five per boring	See Worksheet #14
KCH06-SB05	Soil	0 to 0.5 4 to 6 8 to 10 14 to 16 18 to 20	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Up to five per boring	See Worksheet #14
KCH06-TR01	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR02	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR03	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR04	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR05	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14

### SAP Worksheet #18—Location-Specific Sampling Methods/SOP Requirements Table

Sampling Location/ ID Number <sup>a</sup>	Matrix	Depth <sup>b</sup> (feet bgs)	Analytical Group	Sampling Frequency	Number of Samples (Identify Field Duplicates) <sup>d</sup>	Sampling SOP Reference
KCH06-TR06	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR07	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR08	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR09	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR10	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR11	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
KCH06-TR12	Soil	0 to 0.5 4 to 6 8 to 10 <sup>e</sup>	SVOCs, PAHs, OCPs, PCBs, Metals, Hexavalent Chromium, VOCs <sup>c</sup> , TPH-p <sup>c</sup> , TPH-e <sup>c</sup> , Dioxins/Furans <sup>c</sup>	One time	Average of three per trench	See Worksheet #14
SB-10 (existing well)	Water	Approximately 120 to 150	SVOCs, PAHs, OCPs, PCBs, Total and Dissolved Metals, Hexavalent Chromium, VOCs, TPH-p, TPH-e	Two times	Two	See Worksheet #14

### SAP Worksheet #18—Location-Specific Sampling Methods/SOP Requirements Table

Sampling Location/ ID Number <sup>a</sup>	Matrix	Depth <sup>b</sup> (feet bgs)	Analytical Group	Sampling Frequency	Number of Samples (Identify Field Duplicates) <sup>d</sup>	Sampling SOP Reference
B-21 (existing well)	Water	Approximately 120 to 150	SVOCs, PAHs, OCPs, PCBs, Total and Dissolved Metals, Hexavalent Chromium, VOCs, TPH-p, TPH-e	Two times	Two	See Worksheet #14
KCH06-MW01	Water	TBD	SVOCs, PAHs, OCPs, PCBs, Total and Dissolved Metals, Hexavalent Chromium, VOCs, TPH-p, TPH-e	Two times	Two	See Worksheet #14
KCH06-MW02	Water	TBD	SVOCs, PAHs, OCPs, PCBs, Total and Dissolved Metals, Hexavalent Chromium, VOCs, TPH-p, TPH-e	Two times	Two	See Worksheet #14

**Notes:**

<sup>a</sup> Sample IDs will be sequential numbers (e.g., KCH06-001, KCH06-002, KCH06-003, etc.), which will be logged in a dedicated field logbook to correspond to the location ID, matrix, depth, date, time of collection, and laboratory analyses (See Worksheet #27). Applicable field QC samples also will be included in the sequential numbering. The sample IDs have not yet been assigned.

<sup>b</sup> Soil sampling depth may be adjusted in the field based on change in lithology, PID readings, or field geologist discretion. However, the middle sample depth in the 0- to 10-foot interval will not exceed 6 feet bgs, and the bottom sample depth in the 0- to 10-foot interval will not exceed 10 feet bgs.

<sup>c</sup> VOCs, TPH-p, TPH-e, and dioxins/furans may be analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations, if there is visible contamination, odors, or PID readings in excess of 5 ppm, or visible evidence of burning (dioxins/furans).

<sup>d</sup> Field duplicates for soil will not be collected during this investigation because of the heterogeneity of soil; however, field duplicates for groundwater will be collected at a minimum frequency of 1 per 10 field samples or less.

<sup>e</sup> Depth of soil samples collected in the trench are estimated at up to three sample intervals per trench.

TBD = to be determined upon well installation

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## SAP Worksheet #19 — Field Sampling Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (Number, Size, and Type)	Sample Volume (units)	Preservation Requirements (Chemical, Temperature, Light Protected)	Maximum Holding Time
Water	VOCs	USEPA Method 8260B/5030B EMAX-8260	3 x 40 mL vials	40 mL	HCl; Cool to ≤6°C, not frozen, no headspace	14 days
Water	SVOCs	USEPA Method 8270C/3520C EMAX-8270	2 x 1L amber	1 L	Cool to ≤6°C, not frozen	7 days extract/40 days analysis
Water	PAHs	USEPA Method 8270C SIM/ 3520C EMAX-8270SIM	2 x 1L amber	1 L	Cool to ≤6°C, not frozen	7 days extract/40 days analysis
Water	TPH-p	USEPA Method 8015B/5030B EMAX-8015	3 x 40 mL vials	40 mL	HCl; Cool to ≤6°C, not frozen, no headspace	14 days
Water	TPH-e	USEPA Method 8015B/3520C EMAX-8015	2 x 1L amber	1 L	Cool to ≤6°C, not frozen	7 days extract/40 days analysis
Water	OCPs	USEPA Method 8081A EMAX-8081	2 x 1L amber	1L	Cool to ≤6°C, not frozen	7 days extract/40 days analysis
Water	PCBs	USEPA Method 8082/3520C EMAX-8082	2 x 1L amber	1 L	Cool to ≤6°C, not frozen	7 days extract/40 days analysis
Water	Total Metals	USEPA Method 6020A/7470A EMAX-6020/EMAX-7470	1 x 250 mL polyethylene	100 mL	HNO <sub>3</sub> ; Cool to ≤6°C, not frozen	6 months (28 days mercury)
Water	Dissolved Metals	USEPA Method 6020A/7470A EMAX-6020/EMAX-7470	1 x 250 mL polyethylene	100 mL	Filter; HNO <sub>3</sub> ; Cool to ≤6°C, not frozen	6 months (28 days mercury)
Water	Hexavalent Chromium	USEPA Method 218.6/ EMAX-218.6	1 x 125 mL polyethylene	50 mL	Filter; Ammonium Hydroxide and Ammonium Sulfate buffer to pH 9.0-9.5; Cool to ≤6°C, not frozen	28 days
Soil	VOCs	USEPA Method 8260B/5035 EMAX-8260	3 x EnCore	5 g	Cool to ≤6°C	<b>48 hours</b> to preservation or freezing/14 days to analysis (if frozen or preserved)

## SAP Worksheet #19 — Field Sampling Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (Number, Size, and Type)	Sample Volume (units)	Preservation Requirements (Chemical, Temperature, Light Protected)	Maximum Holding Time
Soil	TPH-p	USEPA Method 8015B/5035 EMAX-8015	2 x EnCore	5 g	Cool to ≤6°C	<b>48 hours</b> to preservation or freezing/14 days to analysis (if frozen or preserved)
Soil	SVOCs	USEPA Method 8270C/3550B EMAX-8270	1 x 8 oz glass	30 g	Cool to ≤6°C, not frozen	14 days extract/ 40 days analysis
Soil	PAHs	USEPA Method 8270C-SIM/ 3550B EMAX-8270SIM		30 g		14 days extract/ 40 days analysis
Soil	TPH-e	USEPA Method 8015B/3550B EMAX-8015		10 g		14 days extract/ 40 days analysis
Soil	OCPs	USEPA Method 8081A EMAX-8081		30 g		14 days extract/ 40 days analysis
Soil	PCBs	USEPA Method 8082/3550B EMAX-8082		30 g		14 days extract/ 40 days analysis
Soil	Total Metals	USEPA Method 6020A/7471A EMAX-6020/EMAX-7471	1 x 8 oz glass	1 g	Cool to ≤6°C, not frozen	180 days (28 days mercury)
Soil	Hexavalent Chromium	USEPA Method 7199 EMAX-7199		10 g		30 days extract/ 7 days analysis
Soil	Dioxins/Furans	USEPA Method 8290A HPL8290	1 x 4 oz glass	10 g	Cool ≤ 6°C, not frozen	30 days extract/ 45 days analysis

Notes:

g = gram  
 HCl = hydrochloric acid  
 HNO<sub>3</sub> = nitric acid  
 L = liter  
 mL = milliliter  
 oz = ounce

### SAP Worksheet #20—Field Quality Control Sample Summary Table

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs	Number of Source Water Blanks	Number of Equipment Blanks <sup>a</sup>	Number of Trip Blanks <sup>a</sup>	Number of PT Samples	Total Number of Samples to Laboratory
Water	VOCs	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	2	NA	19
Water	SVOCs	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	PAHs	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	TPH-p	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	2	NA	19
Water	TPH-e	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	OCPs	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	PCBs	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	Total Metals	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Water	Dissolved Metals	8	2 <sup>b</sup>	2/2 <sup>b</sup>	NA	NA	NA	NA	14
Water	Hexavalent Chromium	8	2 <sup>b</sup>	2/2 <sup>b</sup>	1	2	NA	NA	17
Soil	VOCs	25 <sup>c</sup>	NA	2/2	1	4	4	NA	38
Soil	SVOCs	100	NA	5/5	1	8	NA	NA	119
Soil	PAHs	100	NA	5/5	1	8	NA	NA	119
Soil	TPH-p	25 <sup>c</sup>	NA	2/2	1	4	4	NA	38
Soil	TPH-e	25 <sup>c</sup>	NA	2/2	1	4	NA	NA	34
Soil	OCPs	100	NA	5/5	1	8	NA	NA	119
Soil	PCBs	100	NA	5/5	1	8	NA	NA	119
Soil	Total Metals	100	NA	5/5	1	8	NA	NA	119
Soil	Hexavalent Chromium	100	NA	5/5	1	8	NA	NA	119
Soil	Dioxins/Furans	25 <sup>c</sup>	NA	2/2	1	4	NA	NA	34

Notes:

<sup>a</sup> The number of equipment rinsates and trip blanks is estimated. The actual number of equipment rinsates will depend on number of days in the field where a crew is using nondedicated sampling equipment. The actual number of trip blanks will depend on number of coolers shipped containing samples for VOC to TPH-p analysis.

<sup>b</sup> Two groundwater sampling events will occur. Field duplicates and MS/MSDs will be collected during each sampling event.

<sup>c</sup> VOCs, TPH-p, TPH-e, and dioxins/furans may be analyzed at approximately 25 percent (and a minimum of 10 percent) of the soil sample locations, if there is visible contamination, odors, or PID readings in excess of 5 ppm, or visible evidence of burning (dioxins/furans).

PT = proficiency testing

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## SAP Worksheet #21—Project Sampling SOP References Table

Reference Number	Title, Revision Date, and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP 1	Soil Sampling, 12/19/2014	KCH	PID	N	
SOP 5	Logging of Soil Borings, 12/19/14	KCH	NA	N	
SOP 9	Calibration and Measurement with Field Instruments, 12/19/14	KCH	Water Quality Meter, PID	N	
SOP 11	Decontamination of Personnel and Equipment, 12/19/14	KCH	Buckets	N	
SOP 13	Preparing Field Log Books, 12/19/14	KCH	NA	N	
SOP 16	Chain-of-Custody, 12/19/14	KCH	NA	N	
SOP 18	Equipment Blank and Field Blank Preparation, 12/19/14	KCH	NA	N	
SOP 19	Civil Surveying, 12/19/14	KCH	GPS	N	
SOP 21	Utility Location, 12/19/14	KCH	See Section 14.3	N	
SOP 23	Trenching for Landfill Delineation, 12/19/14	KCH	Excavator	N	

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### SAP Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
PID	Calibration Check	Daily (prior to field use)	Detector resolution limits	Follow procedure as outlined in the manufacturer's instruction manual or contact vendor technical support	Field Manager or designee	Manufacturer's instruction manual and KCH SOP 9 (Worksheet #21)	If equipment is deemed inoperable or malfunctioning, it will be removed from use and replaced.
	Maintenance	Daily cleaning during field use; proper storage during inactive periods	Parameter readings as determined during calibration (above)	Follow procedure as outlined in the manufacturer's water quality meter instruction manual or contact vendor technical support	Field Manager or designee	Manufacturer's instruction manual	If equipment is deemed inoperable or malfunctioning, it will be removed from use and replaced.
Water Quality Meter and Turbidity Meter	Calibration Check	Daily (prior to field use)	Per manufacturer's instructions	Follow procedure as outlined in the manufacturer's instruction manual or contact vendor technical support	Field Manager or designee	Manufacturer's instruction manual and KCH SOP 9 (Worksheet #21)	If equipment is deemed inoperable or malfunctioning, it will be removed from use and replaced.
	Maintenance	Daily cleaning during field use; proper storage during inactive periods	Parameter readings as determined during calibration (above)	Follow procedure as outlined in the manufacturer's water quality meter instruction manual or contact vendor technical support	Field Manager or designee	Manufacturer's instruction manual	If equipment is deemed inoperable or malfunctioning, it will be removed from use and replaced.

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### SAP Worksheet #23—Analytical SOP References Table

Lab SOP Number	Title and Revision Date or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
EMAX-8260	Volatile Organic Compounds by GC/MS, Revision 10, June 6, 2014 – Last Review June 4, 2015	Definitive	Water/Soil – VOCs	GC/MS	EMAX	N
EMAX-8270	SVOCs by GC/MS, Revision 6, July 10, 2014	Definitive	Water/Soil – SVOCs	GC/MS	EMAX	N
EMAX-8270SIM	Semivolatile Organics by GC/MS SIM, Revision 2, July 5, 2011 – Last Review June 16, 2015	Definitive	Water/Soil – PAHs	GC/MS SIM	EMAX	N
EMAX-8015G	Gasoline-Range Organics (GRO), Revision 5, January 24, 2014 – Last Review February 11, 2015	Definitive	Water/Soil – TPH-p	GC	EMAX	N
EMAX-8015D	Diesel-Range Organics (DRO), Revision 6, September 30, 2013 – Last Review September 24, 2014	Definitive	Water/Soil – TPH-e	GC	EMAX	N
EMAX-8081	Organochlorine Pesticides by Gas Chromatography, Revision 8, June 16, 2014 – Last Review June 4, 2015	Definitive	Water/Soil – OCPs	GC	EMAX	N
EMAX-8082	Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 5, July 2, 2014	Definitive	Water/Soil – PCBs	GC	EMAX	N
EMAX-6020	Trace Metals by ICP-MS, Revision 8, June 27, 2013 – Last Review July 8, 2014	Definitive	Water/Soil - Metals	ICP-MS	EMAX	N
EMAX-7470	Mercury in Liquid Waste, Revision 7, December 12, 2012 – Last Review July 8, 2014	Definitive	Water – Mercury	CVAA	EMAX	N

### SAP Worksheet #23—Analytical SOP References Table

Lab SOP Number	Title and Revision Date or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
EMAX-7471	Mercury in Solid or Semisolid Waste, Revision 7, December 12, 2012 – Last Review July 8, 2014	Definitive	Soil - Mercury	CVAA	EMAX	N
EMAX-218.6	Hexavalent Chromium, Revision 5, February 28, 2011 – Last Review June 19, 2015	Definitive	Water – Hexavalent Chromium	IC	EMAX	N
EMAX-7199	Hexavalent Chromium, Revision 3, November 20, 2012 – Last Review February 11, 2015	Definitive	Soil – Hexavalent Chromium	IC	EMAX	N
HPL8290	Instrumental analysis of PCDD and PCDF by HRGS-HRMS (USEPA Method 8290), Revision 11, July 23, 2014	Definitive	Soil – Dioxins/Furans	HRGC HRMS	APPL	N

**Notes:**

Laboratory SOPs meet DoD QSM Version 5.0 requirements. Laboratory SOPs are provided in Attachment 7.

CVAA = cold vapor atomic absorption

GC = gas chromatography

GC/MS = gas chromatography/mass spectrometry

HRGC = high resolution gas chromatography

HRMS = high resolution mass spectrometry

IC = ion chromatography

ICP-MS = inductively coupled plasma-mass spectrometry

### SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria <sup>a</sup>	Corrective Action <sup>b</sup>	Person Responsible for Corrective Action	SOP Reference
GC	ICAL for all analytes (including surrogates)	At instrument setup and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$ . Option 2: linear least squares regression for each analyte $r^2 \geq 0.99$ . Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$ .	Correct problem then repeat ICAL. Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.	EMAX Laboratory Manager/Analyst	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G DoD QSM 5.0
GC	ICV	Once after each ICAL, analyses of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 20\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	EMAX Laboratory Manager/Analyst	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G DoD QSM 5.0
GC	CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for pesticides multi-component analytes (i.e., toxaphene, chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes within $\pm 20\%$ of true value.	Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV.	EMAX Laboratory Manager/Analyst	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G DoD QSM 5.0
GC	Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be $\leq 15\%$ .	Correct problem, then repeat breakdown checks. No samples shall be run until degradation of DDT and Endrin is each $\leq 15\%$ .	EMAX Laboratory Manager/Analyst	EMAX-8081 DoD QSM 5.0
GC	RT window (position establishment)	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed the initial CCV is used.	NA	EMAX Laboratory Manager/Analyst	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G DoD QSM 5.0
GC	RT window (width)	At method setup and after major maintenance (e.g., column change).	RT width is $\pm 3$ times standard deviation for each analyte RT from the 72-hour study.	NA	EMAX Laboratory Manager/Analyst	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G DoD QSM 5.0
GC/MS	Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to specific ion abundance criteria of bromofluorobenzene and decafluorotriphenylphosphine from method	Retune instrument and verify. No samples shall be analyzed without a valid tune.	EMAX Laboratory Manager/Analyst	EMAX-8260 EMAX-8270 EMAX-8270SIM DoD QSM 5.0
GC/MS	Performance check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 20\%$ for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance check. No samples shall be analyzed until performance check is within criteria. The DDT breakdown and benzidine/pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.	EMAX Laboratory Manager/Analyst	EMAX-8270 DoD QSM 5.0

### SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria <sup>a</sup>	Corrective Action <sup>b</sup>	Person Responsible for Corrective Action	SOP Reference
GC/MS	ICAL for all analytes (including surrogates)	At instrument setup and prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$ . Option 2: linear least squares regression for each analyte $r^2 \geq 0.99$ . Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$ .	Correct problem then repeat ICAL. Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.	EMAX Laboratory Manager/Analyst	EMAX-8260 EMAX-8270 EMAX-8270SIM DoD QSM 5.0
GC/MS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 20\%$ of true value.	Correct problem, rerun ICV. If that fails repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	EMAX Laboratory Manager/Analyst	EMAX-8260 EMAX-8270 EMAX-8270SIM DoD QSM 5.0
GC/MS	CCV	Daily before sample analysis, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within $\pm 20\%$ of true value. All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV.	Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV.	EMAX Laboratory Manager/Analyst	EMAX-8260 EMAX-8270 EMAX-8270SIM DoD QSM 5.0
ICP-MS	Tuning	Prior to ICAL.	Mass calibration $\leq 0.1$ amus from the true value; Resolution $< 0.9$ amu full width at 10 percent peak height.	Retune instrument, then reanalyze tuning solution. No samples shall be analyzed without a valid tune.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	Linear dynamic range or high-level check standard	At initial setup and checked every 6 months with a high standard at upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/verify the linear dynamic range. Data cannot be reported above the calibration range without an established/passing high-level check standard.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	ICAL for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used $r^2 \geq 0.99$ .	Correct problem, then repeat ICAL. Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct problem and rerun ICV. If that fails, repeat ICAL. No samples shall be run until ICAL has passed.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	CCV	After every 10 field samples and end of each analytical sequence.	All reported analytes within $\pm 10\%$ of true value.	Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	Low-level calibration check standard (Low Level ICV)	Daily	All reported analytes within $\pm 20\%$ of true value.	Correct problem and repeat ICAL. No samples shall be analyzed without a valid low-level ICV. ICV should be less than or equal to the LOQ.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0
ICP-MS	ICS (also called spectral interference checks)	After ICAL and prior to sample analysis.	<u>ICS-A</u> : Absolute value of concentration for nonspiked analytes $< LOD$ (unless they are verified trace impurities from one of the spike analytes). <u>ICS-AB</u> : within $\pm 20$ true value.	Terminate analysis; identify and correct problem. Reanalyze ICS and all associated samples.	EMAX Laboratory Manager/Analyst	EMAX-6020 DoD QSM 5.0

### SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria <sup>a</sup>	Corrective Action <sup>b</sup>	Person Responsible for Corrective Action	SOP Reference
CVAA	ICAL for all analyses	Daily ICAL prior to sample analysis.	$r^2 \geq 0.99$	Correct the problem and then repeat ICAL.	EMAX Laboratory Manager/Analyst	EMAX-7470 EMAX-7471 DoD QSM 5.0
CVAA	ICV	Once after each ICAL analysis of a second source standard prior to sample analysis.	All reported analytes within $\pm 10\%$ of true value.	Correct the problem. Rerun ICV. If that fails, then repeat ICAL. No samples shall be run until calibration has been verified with a second source standard.	EMAX Laboratory Manager/Analyst	EMAX-7470 EMAX-7471 DoD QSM 5.0
CVAA	CCV	After every 10 field samples, and at the end of analysis sequence.	All reported analytes within $\pm 10\%$ of true value.	Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV.	EMAX Laboratory Manager/Analyst	EMAX-7470 EMAX-7471 DoD QSM 5.0
IC	ICAL for all analytes	ICAL prior to sample analysis.	$r^2 \geq 0.99$	Correct problem, then repeat ICAL. No samples shall be analyzed until ICAL has passed.	EMAX Laboratory Manager/Analyst	EMAX-7199 EMAX-218.6 DoD QSM 5.0
IC	ICV	Once after each ICAL analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within $\pm 10\%$ of true value	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified.	EMAX Laboratory Manager/Analyst	EMAX-7199 EMAX-218.6 DoD QSM 5.0
IC	CCV	Before sample analysis; after every 10 field samples; and at the end of the analysis sequence.	All reported analytes within established RT windows. All reported analytes within $\pm 10\%$ of true value.	Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV.	EMAX Laboratory Manager/Analyst	EMAX-7199 EMAX-218.6 DoD QSM 5.0
IC	RT window (position establishment)	Once per multi-point calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	EMAX Laboratory Manager/Analyst	EMAX-7199 EMAX-218.6 DoD QSM 5.0
IC	RT window (width)	At method setup and after major maintenance (e.g. column change).	RT width is $\pm 3$ times standard deviation for each analyte RT over a 24-hour period.	NA	EMAX Laboratory Manager/Analyst	EMAX-7199 EMAX-218.6 DoD QSM 5.0
HRGC/HRMS	Resolving power	Prior to ICAL and at the beginning and end of each 12-hour period.	Static resolving power $\geq 10,000$ (10 percent valley) for identified masses.	Retune instrument and verify. Rerun affected samples.	APPL Laboratory Manager/Analyst or certified instrument technician	HPL8290 DoD QSM 5.0
HRGC/HRMS	Performance check	Prior to ICAL or CCV; at the beginning of each 12-hour period during which samples or calibration solutions are analyzed.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers: Resolved with a valley $\leq 25\%$ . Identification of all first and last eluters of the eight homologue RT windows and documentation by labeling on the chromatogram. Absolute RTs for switching from one homologous series to the next $\geq 10$ seconds for all components of the mixture.	Correct problem, then repeat column performance check. No sample shall be run until column performance check has passed.	APPL Laboratory Manager/Analyst or certified instrument technician	HPL8290 DoD QSM 5.0

### SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria <sup>a</sup>	Corrective Action <sup>b</sup>	Person Responsible for Corrective Action	SOP Reference
HRGC/HRMS	ICAL for all analytes identified in method	At instrument setup and after ICV or CCV failure, prior to sample analysis, and when a new lot is used as standard source for HRCC-3, sample fortification (IS), or recovery solutions.	Ion abundance ratios in accordance with the method, <u>and</u> signal-to-noise ratio $\geq 10$ for all target analyte ions, <u>and</u> RSD $\leq 20\%$ for the RF for all 17 unlabeled standards; <u>and</u> RSD $\leq 20\%$ for the RFs for the 9 labeled IS.	Correct problem, then repeat ICAL. No samples shall be analyzed until ICAL has passed.	APPL Laboratory Manager/Analyst or certified instrument technician	HPL8290 DoD QSM 5.0
HRGC/HRMS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Ion abundance specified in the method must be met; for unlabeled standards, RF within $\pm 20\%$ of RF established in ICAL; and for labeled standards, RF within $\pm 30\%$ of RF established in ICAL.	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	APPL Laboratory Manager/Analyst or certified instrument technician	HPL8290 DoD QSM 5.0
HRGC/HRMS	CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance specified in the method must be met; for unlabeled standards, RF within $\pm 20\%$ of RF established in ICAL; and for labeled standards, RF within $\pm 30\%$ of RF established in ICAL.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take CAs and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. <u>End-of-run CCV</u> : If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ RPD and the RF established in the ICAL, the mean RF from the two daily CCVs must be used for quantitation of affected samples instead of the ICAL mean RF value. If the starting and ending CCV RFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new initial calibration if it is analyzed within 2 hours. Otherwise analyze samples with positive detections, if necessary. Results may not be reported without a valid CCV.	APPL Laboratory Manager/Analyst or certified instrument technician	HPL8290 DoD QSM 5.0

Notes:

<sup>a</sup> Instruments will be calibrated and acceptance criteria met before sample analysis, and calibration will be performed consistent with the DoD QSM Version 5.0 (DoD, 2013) and published USEPA methods.

<sup>b</sup> No averaging or manual integration will be permitted for instrument calibration or method/QC compliance.

%D = percent difference  
 amu = atomic mass unit  
 CCV = continuing calibration verification  
 ICAL = initial calibration  
 ICS = interference check solution

ICV = initial calibration verification  
 IS = internal standard  
 RPD = relative percent difference  
 RSD = relative standard deviation  
 RF = response factor  
 RT = retention time

**SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table**

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC GC/MS	Change syringe needles/syringes	Instrument performance checks	Visually inspect for dirt or deterioration	Every 3 months	GC: CCV pass GC/MS: tune and CCV pass	Replace syringe or syringe needle	EMAX Analyst or certified instrument technician	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G EMAX-8260 EMAX-8270 EMAX-8270SIM
GC GC/MS	Gas drying and purifying cartridges	Instrument performance checks	Visually inspect for dirt or deterioration	Every 6 to 12 months	Lack of moisture; CCV pass	Replace cartridge	EMAX Analyst or certified instrument technician	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G EMAX-8260 EMAX-8270 EMAX-8270SIM
GC GC/MS	Injection port maintenance	Instrument performance checks	Liner insert, O-rings, septa, column clip	Weekly for liner, monthly for O-rings, daily for septa, or when instrument performance declines	Restoration of operational parameters. GC/MS: tune and CCV pass GC-FID: CCV pass	Replacement of internal components as needed	EMAX Analyst or certified instrument technician	EMAX-8081 EMAX-8082 EMAX-8015D EMAX-8015G EMAX-8260 EMAX-8270 EMAX-8270SIM
GC/MS	Detector maintenance	Column change, failing tune	Change detector or pump oil	Oil level and quality visually examined monthly; oil physically changed every 6 months	Tune passes; scan does not indicate presence of air or water	Refill or exchange the oil; clean parts; reanalyze tune	EMAX Analyst or certified instrument technician	EMAX-8260 EMAX-8270 EMAX-8270SIM
ICP-MS/CVAA	Check instrument connections, gas flow, pressure	Leak test	Visually inspect for wear or damage	Daily or when instrument performance declines	Restoration of operational parameters; CCV pass	Request service	EMAX Analyst or certified instrument technician	EMAX-6020 EMAX-7470 EMAX-7471
ICP-MS	Nebulizer, lens voltage, cone	Instrument performance checks	Visually inspect for dirt or deterioration	Daily	Restoration of operational parameters; CCV pass	Rerun ICV or CCV	EMAX Analyst or certified instrument technician	EMAX-6020
ICP-MS	Filters, torch	Instrument performance, failing ICV or CCV	Visually inspect for dirt or deterioration	Monthly	Restoration of operational parameters; ICV or CCV pass	Rerun ICV or CCV	EMAX Analyst or certified instrument technician	EMAX-6020
IC	Visual inspection	Inspection	Check pump for leaks or spills and visually inspect air lines for crimping or discoloration	Daily	Restoration of operational parameters	Isolate and repair leaks; replace damaged lines	EMAX Analyst or certified instrument technician	EMAX-7199 EMAX-218.6

**SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table**

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
IC	Parameter setup	Physical check	Check pressure, effluent, and detector, and make sure flow rate is set per SOP	Prior to use	Restoration of operational parameters	Note all actions in Daily Maintenance Log	EMAX Analyst or certified instrument technician	EMAX-7199 EMAX-218.6
HRGC/HRMS	Parameter setup	Physical check	Physical check	Initially, prior to daily continuing calibration	All parameters must match those set in the SOP	If parameters do not match the SOP, reset them to proper values	APPL Analyst or certified instrument technician	HPL8290
HRGC/HRMS	Resolution check	Instrument performance	Conformance to instrument tuning	Initially, prior to acquisition and following acquisition	10,000 amu requirement	Correct the problem and repeat resolution check	APPL Analyst or certified instrument technician	HPL8290
HRGC/HRMS	Replace capillary inserts and septa	Physical check	Look for chromatograms exhibiting peak tailing	At least once a week or for every 100 injections or as needed	NA	NA	APPL Analyst or certified instrument technician	HPL8290
HRGC/HRMS	Clean the split seal and injector ports	Physical check	Look for reduced peak size or breakdown peaks	As needed	NA	NA	APPL Analyst or certified instrument technician	HPL8290

Note:  
 FID = flame ionization detector

## SAP Worksheet #26—Sample Handling System

<b>Sample Collection, Packaging, and Shipment</b>
Sample Collection (Personnel/Organization): Sampling field personnel/KCH
Sample Packaging (Personnel/Organization): Sample Management Coordinator/KCH
Coordination of Shipment (Personnel/Organization): Sample Management Coordinator/KCH
Type of Shipment/Carrier: Laboratory courier or FedEx (or equivalent)
<b>Sample Receipt and Analysis</b>
Sample Receipt (Personnel/Organization): Laboratory-designated sample custodians/EMAX, APPL
Sample Custody and Storage (Personnel/Organization): Laboratory-designated sample custodians/EMAX, APPL
Sample Preparation (Personnel/Organization): Laboratory sample preparation personnel/EMAX, APPL
Sample Determinative Analysis (Personnel/Organization): Laboratory analytical chemists/EMAX, APPL
<b>Sample Archiving</b>
Field Sample Storage (number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (number of days from extraction/digestion): 40 days after extraction/digestion
Biological Sample Storage (number of days from sample collection): Not applicable—no biological samples will be collected
<b>Sample Disposal</b>
Personnel/Organization: Laboratory Waste Disposal Coordinator/Laboratory-designated sample custodians/EMAX, APPL
Number of Days from Analysis: 60 days after final sample results are reported, unless there is a hold on a particular sample or previous arrangements have been made

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## SAP Worksheet #27—Sample Custody Requirements

### Sample Identification Procedures

Each soil and groundwater sample will be given a unique, sequential ID number that is blind to the laboratory and carried through from sample collection to data reporting. Samples will be assigned an alpha-numeric identifier that will be tied to the sampling location and sampling depth through a separate logbook that will be maintained in the field by field sampling personnel. The field sampling personnel’s logbook will be kept in addition to the COC record in the project files.

The sequential numbering will begin at KCH06-001, KCH06-002, KCH06-003, and so on, until all samples are collected. The sequential numbers will also be applied to field QC samples. As an example, the following information will be recorded in a table in field sampling personnel’s logbook, for each sample:

**Example:**

Sequential ID on Label and COC	Date	Time	Location	Sample Depth (feet bgs)	Matrix	Sample Type
KCH006-001	10/9/14	0845	KCH06-SB01	0 to 0.5	Soil	N
KCH006-002	10/9/14	0900	KCH06-SB01	4 to 6	Soil	N
etc...						
KCH006-010	10/9/14	0800	QC-TB	NA	Water	TB
KCH006-011	10/9/14	1700	QC-EB	NA	Water	EB

For Sample Type:  
 N = normal sample  
 TB = trip blank  
 EB = equipment blank

Sequential IDs will be populated on the table in the field logbook and on waterproof sample labels as described in Worksheet #14 and KCH SOP 13, Preparing Field Log Books (Attachment 3). The table will be copied or scanned daily and submitted to the Data Manager with copies of the COC records. The original table will be maintained in the field until sampling is complete.

### Field Sample Handling and Custody Procedures

Field sample custody procedures include sample collection, packaging, shipment, and delivery to the laboratory. Custody of field samples will be maintained, and custody transfer will be documented from the time of sample collection through receipt of samples at the analytical laboratory, using COC and custody seal procedures. These requirements will be fulfilled by the KCH field sampling personnel. Each sample will be considered to be in the field personnel’s custody if any one of the following is true:

- The sample is in the person’s physical possession.
- The sample is in view of the person after that person has taken possession.
- The sample is secured so that no one can tamper with the sample.
- The sample is secured in an area that is restricted to authorized personnel.

Field samples will be handled and prepared in the field for submittal to the analytical laboratory for analysis. Field personnel will use the following procedures when packing and transporting samples to the laboratory:

- Check samples for proper labeling and sample information.
- Waterproof metal or equivalent strength plastic coolers will be used for samples.
- Coolers will be lined with double garbage bags.
- Samples will be placed in resealable plastic bags, and placed inside of the garbage-bag-lined cooler.
- Samples that have similar holding times or special handling requirements will be packaged in the same cooler under the same COC record.
- Glass sample containers will first be wrapped in bubble wrap, then placed in double resealable plastic bags.
- A temperature blank will be placed in the bottom of each cooler with the samples.
- If aqueous or soil samples collected for VOCs or TPH-p analyses are included in a cooler, a trip blank will also be included in the cooler, for each analysis.
- Samples will be checked for proper labeling and sample information.
- Ice will be double-bagged using double resealable plastic bags and placed on top of and between the sample bags, until the cooler is full.
- Paperwork (i.e., associated COC records) will be placed in a double resealable plastic bag and taped to the inside lid of the cooler.
- The cooler will be taped and secured using signed custody seals.
- Signed custody seals will be placed on the front or both sides of the cooler before the custody of the cooler is relinquished to the overnight carrier or courier.

### **Chain-of-Custody Procedures**

The COC record (an example is provided at the end of this worksheet) will document the transfer of sample custody from the time of sample collection to laboratory receipt, and will accompany the samples from the field to the analytical laboratory. Samples will be shipped to the primary analytical laboratory. If necessary, the primary analytical laboratory will then package and ship samples to other pre-approved DoD ELAP-accredited laboratory subcontractors.

When custody of the samples is relinquished from one party to another, the individuals involved will sign, date, and record the time of transfer on the COC record. The COC records may consist of an original top copy and two carbonless copies, or the records may be in a carbonless COC format. When using the carbonless COC format, the original copy will be transmitted to the primary analytical laboratory with the samples. The second copy will be retained in project files for the Field Manager, Project Chemist, and Database Manager. A copy of each COC record will be saved in the project files. Upon transfer of the samples to the

primary analytical laboratory, field personnel will sign and date the COC records. Field personnel will make a copy of the signed COC record and scan a copy of each COC record to be saved electronically in the project files.

The COC record will be completed by each field sampling team using waterproof ink. Corrections will be made with a single line-out, the error will be initialed and dated, and then the correct information will be entered. Empty fields on the COC record will be single line crossed out or "Z'd" out, with the date and signature entered by the field sampling team. If samples are to be delivered to the laboratory by an overnight carrier, the airbill number will be recorded, and the COC records will be placed in a waterproof plastic bag and taped to the inside lid of the sample cooler prior to sealing with appropriate secure tape and custody seals. These requirements will be fulfilled by the KCH field sampling personnel. COC procedures are documented in detail in KCH SOP 16, Chain of Custody (Attachment 3).

### **Custody Seals**

Custody seals will be placed on the outside of each sample cooler so that the seals must be broken to open the sample cooler. After field samples are placed into coolers, two or more custody seals will be placed on the outside of the cooler prior to shipment or transport. Each custody seal will be initialed and dated by the field sampling team and affixed to the cooler, and taped over using clear strapping tape.

### **Laboratory Sample Custody Procedures**

Laboratory sample custody procedures include the receipt of samples, archiving, and disposal. Custody of samples will be maintained, and custody transfer will be documented from the time of sample receipt through sample disposal by the analytical laboratory, consistent with the analytical laboratory's SOPs.

The analytical laboratories will have established custody procedures, which include the following:

- Designation of a sample custodian.
- Completion by the custodian of the COC record, sample tags, and laboratory request sheets, including documentation of sample condition upon receipt.
- Laboratory sample tracking and documentation procedures.
- Secure sample storage in the appropriate environment (e.g., refrigerated, dry), consistent with analytical method requirements.
- Proper data logging and documentation procedures, including custody of original laboratory records.

Upon arrival of the samples at the analytical laboratory, a sample custodian will take custody of the samples, assess the integrity of sample containers, and verify that the information on the sample labels matches the information on the associated COC record. The laboratory will restrict access to the storage areas to authorized laboratory personnel only, to prevent unauthorized contact with samples, extracts, or documentation. The sample custodian will maintain security of the samples in accordance with the analytical laboratory SOP.



## SAP Worksheet #28a—Laboratory QC Samples Table

Matrix: Water/Soil

Analytical Group: VOCs

Analytical Method/ SOP Reference: USEPA Method 8260B/EMAX-8260

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0, Control Limits—see Table 28a-1 If the analytes are not listed, use in- house LCS limits. RPD ≤ 20	Correct the problem. If required, reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0, Control Limits—see Table 28a-1 RPD ≤ 20
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0, Control Limits—see Table 28a-1 If the analytes are not listed, use in- house limits. RPD of all analytes ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28a-1 RPD ≤ 20
Surrogates	All field and QC sample	Reference tables of DoD QSM Version 5.0, Control Limits—see Table 28a-1 If the surrogates are not listed, use in- house surrogate limits.	Correct the problem. If required, reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28a-1
IS	Every field sample, standard, and QC sample	RT within ± 10 seconds from RT of the midpoint in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect the GC/MS for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If CA fails in the field samples, analytes associated with noncompliant IS will be qualified and explained in the case narrative.	EMAX Laboratory Analyst	Precision/Accuracy	RT and EICP

Notes:

EICP = extracted ion current profile  
 LCSD = laboratory control sample duplicate  
 MB = method blank  
 ME = marginal exceedance

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**Table 28a-1 Method/SOP QC Acceptance Limits – VOCs**

<b>VOCs by USEPA Method 8260B</b>		
<b>Analyte</b>	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
Acetone	36 to 164	39 to 160
Benzene	77 to 121	79 to 120
Bromobenzene	78 to 121	80 to 120
Bromochloromethane	78 to 125	78 to 123
Bromodichloromethane	75 to 127	79 to 125
Bromoform	67 to 132	66 to 130
Bromomethane	53 to 143	53 to 141
2-Butanone	51 to 148	56 to 143
n-Butylbenzene	70 to 128	75 to 128
sec-Butylbenzene	73 to 126	77 to 126
tert-Butylbenzene	73 to 125	78 to 124
Carbon disulfide	63 to 132	64 to 133
Carbon tetrachloride	70 to 135	72 to 136
Chlorobenzene	79 to 120	82 to 118
Chloroethane	59 to 139	60 to 138
Chloroform	78 to 123	79 to 124
Chloromethane	50 to 136	50 to 139
2-Chlorotoluene	75 to 122	79 to 122
4-Chlorotoluene	72 to 124	78 to 122
Dibromochloromethane	74 to 126	74 to 126
1,2-Dibromo-3-chloropropane	61 to 132	62 to 128
1,2-Dibromoethane	78 to 122	77 to 121
Dibromomethane	78 to 125	79 to 123
1,2-Dichlorobenzene	78 to 121	80 to 119
1,3-Dichlorobenzene	77 to 121	80 to 119
1,4-Dichlorobenzene	75 to 120	79 to 118
Dichlorodifluoromethane	29 to 149	32 to 152
1,1-Dichloroethane	76 to 125	77 to 125
1,2-Dichloroethane	73 to 128	73 to 128
1,1-Dichloroethene	70 to 131	71 to 131
cis-1,2-Dichloroethene	77 to 123	78 to 123
trans-1,2-Dichloroethene	74 to 125	75 to 124
1,2-Dichloropropane	76 to 123	78 to 122
1,3-Dichloropropane	77 to 121	80 to 119
2,2-Dichloropropane	67 to 133	60 to 139
1,1-Dichloropropene	76 to 125	79 to 125
cis-1,3-Dichloropropene	74 to 126	75 to 124
trans-1,3-Dichloropropene	71 to 130	73 to 127
Ethylbenzene	76 to 122	79 to 121

**Table 28a-1 Method/SOP QC Acceptance Limits – VOCs**

<b>VOCs by USEPA Method 8260B</b>		
<b>Analyte</b>	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
Hexachlorobutadiene	61 to 135	66 to 134
2-Hexanone	53 to 145	57 to 139
Isopropylbenzene	68 to 134	72 to 131
p-Isopropyltoluene	73 to 127	77 to 127
Methylene chloride	70 to 128	74 to 124
4-Methyl-2-pentanone	65 to 135	67 to 130
Methyl tertiary butyl ether	73 to 125	71 to 124
Tertiary butyl alcohol	68 to 133	68 to 129
n-Propylbenzene	73 to 125	76 to 126
Styrene	76 to 124	78 to 123
1,1,1,2-Tetrachloroethane	78 to 125	78 to 124
1,1,2,2-Tetrachloroethane	70 to 124	71 to 121
Tetrachloroethene	73 to 128	74 to 129
Toluene	77 to 121	80 to 121
1,2,3-Trichlorobenzene	66 to 130	69 to 129
1,2,4-Trichlorobenzene	67 to 129	69 to 130
1,1,1-Trichloroethane	73 to 130	74 to 131
1,1,2-Trichloroethane	78 to 121	80 to 119
Trichloroethene	77 to 123	79 to 123
Trichlorofluoromethane	62 to 140	65 to 141
1,2,3-Trichloropropane	73 to 125	73 to 122
1,2,4-Trimethylbenzene	75 to 123	76 to 124
1,3,5-Trimethylbenzene	73 to 124	75 to 124
m,p-Xylenes	77 to 124	80 to 121
o-Xylene	77 to 123	78 to 122
Naphthalene	62 to 129	61 to 128
Vinyl chloride	56 to 135	58 to 137
<b>Surrogate</b>	<b>%R</b>	
Toluene-D8	85 to 116	89 to 112
4-Bromofluorobenzene	79 to 119	85 to 114
1,2-Dichloroethane-D4	71 to 136	81 to 118
Dibromofluoromethane	78 to 119	80 to 119

**Notes:**

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control limit was used.  
 %R = percent recovery

## SAP Worksheet #28b—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: SVOCs**

**Analytical Method/SOP Reference: USEPA Method 8270C/EMAX-8270**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater. Common laboratory contaminants must not be detected > LOQ.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1 If the analytes are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 20	Correct the problem. If required, reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1 RPD ≤ 20
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1 If the analytes are not listed, use in-house limits if project limits are not specified. RPD of all analytes ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/ Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1 RPD ≤ 20
Surrogates	All field and QC samples	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1; otherwise, use in-house surrogate limits.	Correct the problem, then reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28b-1
IS	Every field sample, standard, and QC sample	RT within ± 10 seconds from RT of the midpoint in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect the GC/MS for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If CA fails in the field samples, analytes associated with noncompliant IS will be qualified and explained in the case narrative.	EMAX Laboratory Analyst	Precision/Accuracy	RT and EICP

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**Table 28b-1 Method/SOP QC Acceptance Limits – SVOCs**

SVOCs by USEPA Method 8270C		
QC Parameters	Soil Control Limits (%R)	Water Control Limits (%R)
1,2,4-Trichlorobenzene	34 to 118	29 to 116
1,2-Dichlorobenzene	33 to 117	32 to 111
1,3-Dichlorobenzene	30 to 115	28 to 110
1,4-Dichlorobenzene	31 to 115	29 to 112
2,4,5-Trichlorophenol	41 to 124	53 to 123
2,4,6-Trichlorophenol	39 to 126	50 to 125
2,4-Dichlorophenol	40 to 122	47 to 121
2,4-Dimethylphenol	30 to 127	31 to 124
2,4-Dinitrophenol	15 to 130 <sup>a</sup>	23 to 143
2,4-Dinitrotoluene	46 to 126	57 to 128
2,6-Dinitrotoluene	46 to 124	57 to 124
2-Chloronaphthalene	41 to 114	40 to 116
2-Chlorophenol	34 to 121	38 to 117
2-Methylnaphthalene	38 to 122	40 to 121
2-Methylphenol	32 to 122	30 to 117
2-Nitroaniline	44 to 127	55 to 127
2-Nitrophenol	36 to 123	47 to 123
2,2-oxybis(1-chloropropane)	33 to 131	37 to 130
3,3'-Dichlorobenzidine	22 to 121	27 to 129
3-Nitroaniline	33 to 119	41 to 128
4,6-Dinitro-2-methylphenol	29 to 132	44 to 137
4-Bromophenyl phenyl ether	46 to 124	55 to 124
4-Chloro-3-methylphenol	45 to 122	52 to 119
4-Chloroaniline	17 to 106	33 to 117
4-Chlorophenyl phenyl ether	45 to 121	53 to 121
4-Methylphenol	42 to 126	25 to 120
4-Nitroaniline	35 to 115 <sup>a</sup>	35 to 120 <sup>a</sup>
4-Nitrophenol	30 to 132	0 to 125 <sup>a</sup>
Acenaphthene	40 to 123	47 to 122
Acenaphthylene	32 to 132	41 to 130
Anthracene	47 to 123	57 to 123
Benzo (a) anthracene	49 to 126	58 to 125
Benzo (a) pyrene	45 to 129	54 to 128
Benzo (b) fluoranthene	45 to 132	53 to 131
Benzo (g,h,i) perylene	43 to 134	50 to 134
Benzo (k) fluoranthene	47 to 132	57 to 129
Benzoic acid	0 to 110 <sup>a</sup>	0 to 125 <sup>a</sup>
Benzyl alcohol	29 to 122	31 to 112
Bis (2-chloroethoxy) methane	36 to 121	48 to 120
Bis (2-chloroethyl) ether	31 to 120	43 to 118

**Table 28b-1 Method/SOP QC Acceptance Limits – SVOCs**

SVOCs by USEPA Method 8270C		
QC Parameters	Soil Control Limits (%R)	Water Control Limits (%R)
Bis (2-ethylhexyl) phthalate	51 to 133	55 to 135
Butyl benzylphthalate	48 to 132	53 to 134
Carbazole	50 to 123	60 to 122
Chrysene	50 to 124	59 to 123
Dibenz(a,h) anthracene	45 to 134	51 to 134
Dibenzofuran	44 to 120	53 to 118
Diethyl phthalate	50 to 124	56 to 125
Dimethyl phthalate	48 to 124	45 to 127
Di-n-butylphthalate	51 to 128	59 to 127
Di-n-octylphthalate	45 to 140	51 to 140
Fluoranthene	50 to 127	57 to 128
Fluorene	43 to 125	52 to 124
Hexachlorobenzene	45 to 122	53 to 125
Hexachlorobutadiene	32 to 123	22 to 124
Hexachloroethane	28 to 117	21 to 115
Indeno (1,2,3-c,d) pyrene	45 to 133	52 to 134
Isophorone	30 to 122	42 to 124
Naphthalene	35 to 123	40 to 121
Nitrobenzene	34 to 122	45 to 121
n-Nitrosodimethylamine	23 to 120	25 to 110 <sup>a</sup>
n-Nitrosodi-n-propylamine	36 to 120	49 to 119
n-Nitrosodiphenylamine	38 to 127	51 to 123
Pentachlorophenol	25 to 133	35 to 138
Phenanthrene	50 to 121	59 to 120
Phenol	34 to 121	0 to 115 <sup>a</sup>
Pyrene	47 to 127	57 to 126
<b>Surrogate</b>	<b>%R</b>	
2-Fluorobiphenyl	44 to 115	44 to 119
2-Fluorophenol	35 to 115	19 to 119
Nitrobenzene-d5	37 to 122	44 to 120
2,4,6-Tribromophenol	39 to 132	43 to 140
Terphenyl-d14	54 to 127	50 to 134
Phenol-d5	33 to 122	10 to 115 <sup>a</sup>

Notes:

The accuracy and precision limits are based on those in the DoD QSM, Version 5.0 (DoD, 2013).

<sup>a</sup> If a DoD QSM Version 5.0 control limit was not available, either the DoD QSM Version 4.2 (DoD, 2010) or the laboratory-generated control limit was used.

## SAP Worksheet #28c—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: PAHs**

**Analytical Method/ SOP Reference: USEPA Method 8270CSIM/EMAX-8270SIM**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1 If the analytes are not listed, use in- house LCS limits if project limits are not specified. RPD ≤ 20	Correct the problem. If required, reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1 RPD ≤ 20
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1 If the analytes are not listed, use in- house limits if project limits are not specified. RPD of all analytes ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1 RPD ≤ 20
Surrogates	All field and QC sample	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1; otherwise, use in-house surrogate limits.	Correct the problem, then reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28c-1
IS	Every field sample, standard, and QC sample	RT within ± 10 seconds from RT of the midpoint in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect the GC/MS for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If CA fails in the field samples, analytes associated with noncompliant IS will be qualified and explained in case narrative.	EMAX Laboratory Analyst	Precision/Accuracy	RT and EICP

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**Table 28c-1 Method/SOP QC Acceptance Limits – PAHs**

<b>PAHs by USEPA Method 8270CSIM</b>		
<b>Analyte</b>	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
1-methylnaphthalene	43 to 111	41 to 115
2-methylnaphthalene	39 to 114	39 to 114
Acenaphthene	44 to 111	48 to 114
Acenaphthylene	39 to 116	35 to 121
Anthracene	50 to 114	53 to 119
Benzo (a) anthracene	54 to 122	59 to 120
Benzo (a) pyrene	50 to 125	53 to 120
Benzo (b) fluoranthene	53 to 128	53 to 126
Benzo (g,h,i) perylene	49 to 127	44 to 128
Benzo (k) fluoranthene	56 to 123	54 to 125
Chrysene	57 to 118	57 to 120
Dibenz(a,h) anthracene	50 to 129	44 to 131
Fluoranthene	55 to 119	58 to 120
Fluorene	47 to 114	50 to 118
Indeno (1,2,3-c,d) pyrene	49 to 130	48 to 130
Naphthalene	38 to 111	43 to 114
Phenanthrene	49 to 113	53 to 115
Pyrene	55 to 117	53 to 121
<b>Surrogate</b>	<b>%R</b>	
2-Fluorobiphenyl	46 to 115	53 to 106
Nitrobenzene-d5	44 to 125	55 to 111
Terphenyl-d14	58 to 133	58 to 132

Notes:

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control will be used.

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## SAP Worksheet #28d—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: TPH-p, TPH-e**

**Analytical Method/ SOP Reference: USEPA Method 8015B/ EMAX-8015G, EMAX-8015D**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference the DoD QSM Version 5.0, Control Limits for batch control —see Table 28d-1 If the analytes are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30	Correct the problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0, Control Limits—see Table 28d-1 RPD ≤ 30
MS/MSD	One per SDG or every 20 samples or less; all target compounds to be spiked; project designated samples.	Reference the DoD QSM Version 5.0, Control Limits for batch control —see Table 28d-1 If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD of all analytes ≤ 30	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/ Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28d-1 RPD ≤ 30
Surrogates	All field and QC sample	Reference the DoD QSM Version 5.0, Control Limits—see Table 28d-1 or use in-house surrogate limits.	Correct the problem, then reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28d-1

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**Table 28d-1 Method/SOP QC Acceptance Limits – TPH**

TPH by USEPA Method 8015B		
Analyte	Method/SOP QC Acceptance Limits	
QC Parameters	Soil Control Limits (%R)	Water Control Limits (%R)
TPH-p as gasoline	79 to 122	78 to 122
TPH-e as diesel	38 to 132	36 to 132
Surrogates	%R	
4-Bromofluorobenzene (TPH-p)	67 to 134	69 to 133
Bromobenzene <sup>a</sup> (TPH-e)	60 to 130	60 to 130
Hexacosane <sup>a</sup> (TPH-e)	60 to 130	60 to 130

Notes:

The accuracy and precision limits are based on those in the DoD QSM, Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control limit was used.

<sup>a</sup> Bromobenzene and hexacosane were used as surrogates with DoD QSM Version 4.2 (DoD, 2010).

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## SAP Worksheet #28e—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: OCPs**

**Analytical Method/SOP Reference: USEPA Method 8081A/EMAX-8081**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit whichever is greater.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1 If the analytes are not listed, use in- house LCS limits if project limits are not specified. RPD ≤ 30	Correct the problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1 RPD ≤ 30
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1 If the analytes are not listed, use in- house limits if project limits are not specified. RPD of all analytes ≤ 30	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1 RPD ≤ 30
Surrogates	All field and QC sample	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1; otherwise, use in-house surrogate limits.	Correct the problem, then reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28e-1
Confirmation of Positive Results (second column)	All positive results must be confirmed	Calibration and QC criteria for second column are the same as initial or primary column analysis. Results between primary and secondary column ≤ 40%.	If RPD is > 40%, discuss in the case narrative.	EMAX Laboratory Analyst	Precision/Accuracy	RPD < 40%

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**Table 28e-1. Method/SOP QC Acceptance Limits – OCPs**

<b>OCPs by USEPA Method 8081A</b>		
<b>ANALYTE</b>	<b>Method/SOP QC Acceptance Limits</b>	
<b>QC Parameters</b>	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
4,4'-DDD	56 to 139	56 to 143
4,4'-DDE	56 to 134	57 to 135
4,4'-DDT	50 to 141	51 to 143
Aldrin	45 to 136	45 to 134
alpha-BHC	45 to 137	54 to 138
alpha-Chlordane	54 to 133	60 to 129
beta-BHC	50 to 136	56 to 136
delta-BHC	47 to 139	52 to 142
Dieldrin	56 to 136	60 to 136
Endosulfan I	53 to 132	62 to 126
Endosulfan II	53 to 134	52 to 135
Endosulfan sulfate	55 to 136	62 to 133
Endrin	57 to 140	60 to 138
Endrin aldehyde	35 to 137	51 to 132
Endrin ketone	55 to 136	58 to 134
gamma-BHC (Lindane)	49 to 135	59 to 134
gamma-Chlordane	53 to 135	56 to 136
Heptachlor	47 to 136	54 to 130
Heptachlor epoxide	52 to 136	61 to 133
Methoxychlor	52 to 143	54 to 145
<b>Surrogates</b>	<b>%R</b>	
Decachloro Biphenyl (DCB)	55 to 130	30 to 135
Tetrachloro-m-xylene	42 to 129	44 to 124

**Notes:**

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (2013). If a DoD QSM control limit was not available, the laboratory-generated control limit was used.

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## SAP Worksheet #28f—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: PCBs**

**Analytical Method/SOP Reference: USEPA Method 8082/EMAX-8082**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct the problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1 If the analytes are not listed, use in- house LCS limits if project limits are not specified. RPD ≤ 30	Correct the problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1 RPD ≤ 30
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1 If the analytes are not listed, use in- house limits if project limits are not specified. RPD of all analytes ≤ 30	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1 RPD ≤ 30
Surrogates	All field and QC sample	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1; otherwise, use in-house surrogate limits.	Correct the problem, then reprepare and reanalyze all samples with failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met, and explain in case narrative.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28f-1
Confirmation of Positive Results (second column)	All positive results must be confirmed	Calibration and QC criteria for second column are the same as initial or primary column analysis. Results between primary and secondary column ≤ 40%.	If RPD is > 40%, discuss in the case narrative	EMAX Laboratory Analyst	Precision/Accuracy	RPD < 40%

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**Table 28f-1 Method/SOP QC Acceptance Limits – PCBs**

<b>Analyte</b>	<b>Method/SOP QC Acceptance Limits</b>	
	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
Aroclor 1016	47 to 134	46 to 129
Aroclor 1260	53 to 140	45 to 124
<b>Surrogate</b>	<b>%R</b>	
Tetrachloro-m-xylene	44 to 130	60 to 130
Decachlorobiphenyl <sup>a</sup>	60 to 125	40 to 135

Notes:

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control limit was used.

<sup>a</sup>Decachlorobiphenyl was used for the surrogate with DoD Version 4.2 (DoD, 2010) control limits.

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## SAP Worksheet #28g—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: Metals**

**Analytical Method/ SOP Reference: USEPA Method 6020A/7470A/7471A / EMAX-6020, EMAX-7470A, EMAX-7471**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No analytes detected > ½ LOQ; or 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprepare and reanalyze MB and all samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < ½ LOQ
ICB/CCB	Before beginning a sample run, after every 10 field samples, and at end of analysis sequence.	No analytes > LOD	Correct problem and repeat ICAL. All samples following the last acceptable ICB or CCB must be reanalyzed. For CCB, failures due to carryover may not require an ICAL.	EMAX Laboratory Analyst	Accuracy	Detections < LOD
LCS/LCSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28g-1 RPD ≤ 20	Correct problem. If required, reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analyte, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28g-1 RPD ≤ 20
MS/MSD	One per preparatory batch	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28g-1 If the analytes are not listed, use in- house limits. RPD of all analytes ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Reference tables of DoD QSM Version 5.0 Control Limits—see Table 28g-1 RPD ≤ 20
IS	Every field sample and QC sample	IS intensity in the sample within 30% to 120% of intensity of the IS in the ICAL.	If recoveries are acceptable for QC samples but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria are met. For failed QC samples, correct problem, and rerun all associated failed field samples.	EMAX Laboratory Analyst	Precision/ Accuracy	RT and EICP
ICP Serial Dilution	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project. Only applicable for samples with concentrations > 50x LOD (prior to dilution). Use with MS/MSD or PDS data to confirm matrix effect.	EMAX Laboratory Analyst	Accuracy	If original sample result is at least 50 times the instrument DL, five-fold dilution must agree within ±10 percent of the original result
Post-Digestion Spike	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80 to 120%.	Check for instrumental problem then reanalyze post-digestion spike addition if appropriate. Criteria apply for samples with concentrations < 50x LOQ prior to dilution. Flag result as needed.	EMAX Laboratory Analyst	Accuracy	80 to 120 %R of expected results

Notes:

CCB = continuing calibration blank

ICB = initial calibration blank

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**Table 28g-1 Method/SOP QC Acceptance Limits – Metals**

<b>Metals by USEPA Method 6020A, 7471A, and 7470A</b>		
<b>Analyte</b>	<b>Soil Control Limits (%R)</b>	<b>Water Control Limits (%R)</b>
Aluminum	78 to 124	84 to 117
Antimony	72 to 124	85 to 117
Arsenic	82 to 118	84 to 116
Barium	86 to 116	86 to 114
Beryllium	80 to 120	83 to 121
Cadmium	84 to 116	87 to 115
Calcium	86 to 118	87 to 118
Chromium	83 to 119	85 to 116
Cobalt	84 to 115	86 to 115
Copper	84 to 119	85 to 118
Iron	81 to 124	87 to 118
Lead	84 to 118	88 to 115
Magnesium	80 to 123	83 to 118
Manganese	85 to 116	87 to 115
Mercury	74 to 126	82 to 119
Molybdenum	83 to 114	83 to 115
Nickel	84 to 119	85 to 117
Potassium	85 to 119	87 to 115
Selenium	80 to 119	80 to 120
Silver	83 to 118	85 to 116
Sodium	79 to 125	85 to 117
Thallium	83 to 118	82 to 116
Vanadium	82 to 116	86 to 115
Zinc	82 to 119	83 to 119

Notes:

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control limit was used.

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## SAP Worksheet #28h—Laboratory QC Samples Table

**Matrix: Water/Soil**

**Analytical Group: Hexavalent Chromium**

**Analytical Method/SOP Reference: USEPA Method 218.6,7199/EMAX-218.6,EMAX-7199**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparatory batch	No target compounds > 1/2 LOQ; or 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprepare and analyze MB and all samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Results may not be reported without a valid MB.	EMAX Laboratory Analyst	Accuracy	Detections < 1/2 LOQ
LCS/LCSD	One per preparatory batch	Use laboratory in-house limits. RPD ≤ 20	Correct problem. If required, reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analyte, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Results may not be reported without a valid LCS.	EMAX Laboratory Analyst	Accuracy	Use laboratory in-house limits. RPD ≤ 20
MS/MSD	One per preparatory batch	Use laboratory in-house limits. RPD ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	EMAX Laboratory Analyst	Precision/Accuracy	Use laboratory in-house limits. RPD ≤ 20

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## SAP Worksheet #28i—Laboratory QC Samples Table

**Matrix: Soil**

**Analytical Group: Dioxins/Furans**

**Analytical Method/SOP Reference: USEPA Method 8290A/HPL8290**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MB	Once per preparatory blank, run after calibration standards and before samples.	No target compounds > ½ LOQ; or > 1/10 the amount measured in any sample; or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprepare and reanalyze MB and all samples processed with contaminated MB. If reanalysis cannot be performed, qualify the data and explain in case narrative. Results may not be reported without a valid MB.	APPL Laboratory Analyst	Accuracy	Detections < LOD
LCS	One per preparatory batch	Reference the DoD QSM Version 5.0 - See Table 28i-1	Correct problem. If required, reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample volume is available. If cannot reanalyze, flag data and explain in case narrative. Results may not be reported without a valid LCS.	APPL Laboratory Analyst	Accuracy	See Table 28i-1
MS/MSD	One per preparatory batch	Reference the DoD QSM Version 5.0 control limits for batch control - See Table 28i-1 If the analytes are not listed, use in-house limits if project limits are not specified. RPD of all analytes ≤ 20	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	APPL Laboratory Analyst	Precision/Accuracy	See Table 28i-1 RPD ≤ 20
EMPC	Every sample with a response S/N ≥ 2.5 for both quantitation ions	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5.	NA	APPL Laboratory Analyst	Accuracy	S/N ≥ 2.5
IS	Every field sample, standard, and QC sample	See Table 28i-1 40 to 135 %R, prior to dilutions	Correct problem. If required, reprepare and reanalyze the samples with failed IS. Apply Q-flag to results of all affected samples.	APPL Laboratory Analyst	Precision/Accuracy	See Table 28i-1 40 to 135 %R

Notes:  
 S/N = signal to noise

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**Table 28i-1. Method/SOP QC Acceptance Limits – Dioxins/Furans**

<b>Dioxins/Furans by USEPA Method 8290A</b>	
<b>Analyte</b>	<b>Method/SOP QC Acceptance Limits</b>
<b>QC Parameters</b>	<b>Soil Control Limits (%R)</b>
2,3,7,8-TCDD	70 to 128
1,2,3,7,8-PeCDD	74 to 125
1,2,3,4,7,8-HxCDD	72 to 131
1,2,3,6,7,8-HxCDD	74 to 134
1,2,3,7,8,9-HxCDD	71 to 138
1,2,3,4,6,7,8-HpCDD	76 to 125
OCDD	72 to 135
2,3,7,8-TCDF	75 to 135
1,2,3,7,8-PeCDF	77 to 131
2,3,4,7,8-PeCDF	75 to 128
1,2,3,4,7,8-HxCDF	77 to 130
1,2,3,6,7,8-HxCDF	74 to 134
1,2,3,7,8,9-HxCDF	74 to 135
2,3,4,6,7,8-HxCDF	74 to 133
1,2,3,4,6,7,8-HpCDF	73 to 135
1,2,3,4,7,8,9-HpCDF	76 to 125
OCDF	66 to 144
<b>QC Parameters</b>	<b>IS (%R prior to dilution)</b>
13C-2,3,7,8-TCDD	40 to 135
13C -2,3,7,8-TCDF	40 to 135
13C -1,2,3,7,8-PeCDD	40 to 135
13C -1,2,3,7,8-PeCDF	40 to 135
13C -1,2,3,6,7,8-HxCDD	40 to 135
13C -1,2,3,6,7,8-HxCDF	40 to 135
13C -1,2,3,4,6,7,8-HpCDD	40 to 135
13C -1,2,3,4,6,7,8-HpCDF	40 to 135
13C-OCDD	40 to 135

Notes:

The accuracy and precision limits are based on those in the DoD QSM Version 5.0 (DoD, 2013). If a DoD QSM control limit was not available, the laboratory-generated control limit will be used.

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## SAP Worksheet #29—Project Documents and Records Table

Document	Where Maintained*
APP and work plan with SAP	KCH project files and NAVFAC Southwest Administrative Record
Field notes/logbook	KCH project file
COC	KCH project file and analytical laboratory (EMAX, APPL)
Laboratory raw data	Analytical Laboratory, KCH project file, NAVFAC Southwest Administrative Record
Field audit	KCH project file
CA	KCH project file and analytical laboratory (EMAX, APPL)
Laboratory equipment maintenance logs	KCH project file and analytical laboratory (EMAX, APPL)
Sample preparation	KCH project file, analytical laboratory (EMAX, APPL) and NAVFAC Southwest Administrative Record
Run logs	KCH project file, analytical laboratory (EMAX, APPL) and NAVFAC Southwest Administrative Record
Sample disposal	KCH project file and analytical laboratory (EMAX, APPL)
Contract Laboratory Program (CLP)-equivalent (Level IV) analytical laboratory reports, including raw data (report parameters summarized on checklist and provided as attachment)	KCH project file, analytical laboratory, NAVFAC Southwest Administrative Record
Data validation reports	KCH project file, data validation subcontractor (LDC), NAVFAC Southwest Administrative Record

Notes:

\* Files will be stored for a minimum of 7 years (at the KCH San Diego, California office) in accordance with the Comprehensive Long-Term Environmental Action - Navy contract requirement.

Documents submitted to the NAVFAC Southwest Administrative Record will be consistent with NAVFAC Southwest EWI No. 6 (NAVFAC Southwest, 2005).

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### SAP Worksheet #30—Analytical Services Table

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turn-around Time	Laboratory/Organization (name and address, contact person and telephone number)	Backup Laboratory/Organization (name and address, contact person and telephone number)
Soil/Water	VOCs	See Worksheet #18	USEPA 8260B	28 days (calendar)	EMAX Laboratory 1835 W. 205th Street Torrance, California 90501 (310) 618-8889 Contact: Ye Myint	APPL, Inc. 908 North Temperance Avenue Clovis, California 93611 (559) 275-2175 Contact: Cynthia Clark
Soil/Water	SVOCs		USEPA 8270C			
Soil/Water	PAHs		USEPA 8270C SIM			
Soil/Water	TPH-p		USEPA 8015B			
Soil/Water	TPH-e		USEPA 8015B			
Soil/Water	OCPs		USEPA 8081A			
Soil/Water	PCBs		USEPA 8082			
Soil/Water	Metals		USEPA 6020A/ 7470A/7471A			
Soil/Water	Hexavalent Chromium		USEPA 7199/218.6			
Soil	Dioxins/Furans		USEPA 8290A		APPL, Inc. 908 North Temperance Avenue Clovis, California 93611 (559) 275-2175 Contact: Cynthia Clark	ALS-Houston 19408 Park Row, Suite 320 Houston, Texas 77450 (713) 266-1599 Contact: Jim Plassard

**Notes:**

Soil and groundwater samples will be analyzed by laboratories that are certified by the State of California ELAP and accredited by the DoD ELAP (Attachment 8).  
 EMAX DoD ELAP Certification Number L2278, Valid to January 10, 2017  
 APPL DoD ELAP Certification Number L13-238-R2; Valid to November 27, 2015

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### SAP Worksheet #31—Planned Project Assessments Table

<b>Assessment Type</b>	<b>Frequency</b>	<b>Internal or External</b>	<b>Organization Performing Assessment</b>	<b>Person(s) Responsible for Performing Assessment (title and organizational affiliation)</b>	<b>Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)</b>	<b>Person(s) Responsible for Identifying and Implementing CA (title and organizational affiliation)</b>	<b>Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)</b>
Readiness Review	Before mobilizing to the field	Internal	KCH	Health and Safety Manager (KCH)	TOM and Field Manager (KCH)	TOM (KCH)	TOM (KCH)
Field Sampling TSA	Once during sampling	Internal	KCH	TOM (KCH) Program QAM (KCH)	Field Manager (KCH)	Field Manager (KCH)	PQAO (KCH)
Data Review TSA	During field sampling and analysis through validation	Internal	KCH	TOM (KCH) Program QAM (KCH)	Field Manager and Project Chemist (KCH), and EMAX and APPL Laboratory Managers	Project Chemist and Program QAM (KCH), and EMAX and APPL Laboratory Managers	Program QAM and Project Chemist (KCH)

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### SAP Worksheet #32—Assessment Findings and Corrective Action Responses Table

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Readiness Review	Readiness Review Checklist.	Eric Johansen TOM KCH	As soon as possible, within same day of finding	Readiness Review Checklist with outstanding actions completed or addressed prior to project work.	Eric Johansen TOM KCH	1 business day
Field Sampling TSA	Audit form showing results of field audit. If CAs are necessary and cannot be implemented during the audit, these deficiencies will be noted and their resolution will be documented in the CA report.	Jeremiah Stock Field Manager KCH	As soon as possible within same day of finding	Completed Audit Form indicating all CAs taken. Additional documentation will be attached as necessary. Audit form is issued by the Program QAM.	Jeremiah Stock Field Manager KCH	1 business day
		Eric Johansen TOM KCH	1 business day		Eric Johansen TOM KCH	1 business day
		Theresa Rojas Program QAM KCH	1 business day		Theresa Rojas Program QAM KCH	3 business days
		Brenda Reese RPM Navy	1 business day if CA involving more than a 1 day delay is necessary		Brenda Reese RPM Navy	Included with summary report
Data Review TSA	Memo or written audit report.	Theresa Rojas Program QAM KCH	1 business day	Letter or e-mail.	Theresa Rojas Program QAM KCH	3 business days

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**Example of Technical Systems Audit Form**



**TECHNICAL SYSTEMS AUDIT FORM: SOIL BORING/SAMPLING**

Crew Members: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 Boring/Sampling Location: \_\_\_\_\_  
 Work Activity: \_\_\_\_\_

Date: \_\_\_\_\_  
 Time: \_\_\_\_\_

	In Compliance?		If "No" Corrective action taken (Attach additional documentation if necessary)
	Yes	No	
<b>HEALTH &amp; SAFETY</b>			
APP/SSHP readily available	<input type="checkbox"/>	<input type="checkbox"/>	_____
Hard hats	<input type="checkbox"/>	<input type="checkbox"/>	_____
Steel-toed boots	<input type="checkbox"/>	<input type="checkbox"/>	_____
Safety vest	<input type="checkbox"/>	<input type="checkbox"/>	_____
Gloves	<input type="checkbox"/>	<input type="checkbox"/>	_____
Eye protection	<input type="checkbox"/>	<input type="checkbox"/>	_____
Air monitoring conducted in breathing zone	<input type="checkbox"/>	<input type="checkbox"/>	_____
Work area clearly delineated	<input type="checkbox"/>	<input type="checkbox"/>	_____
<b>DOCUMENTATION</b>			
SAP onsite	<input type="checkbox"/>	<input type="checkbox"/>	_____
Boring log	<input type="checkbox"/>	<input type="checkbox"/>	_____
Field notebook	<input type="checkbox"/>	<input type="checkbox"/>	_____
Sample labels	<input type="checkbox"/>	<input type="checkbox"/>	_____
Chain of custody	<input type="checkbox"/>	<input type="checkbox"/>	_____
<b>SOIL BORING</b>			
Hand auger clean	<input type="checkbox"/>	<input type="checkbox"/>	_____
Soil sampler clean	<input type="checkbox"/>	<input type="checkbox"/>	_____
Boring location	<input type="checkbox"/>	<input type="checkbox"/>	_____
Boring log	<input type="checkbox"/>	<input type="checkbox"/>	_____
Total boring depth	<input type="checkbox"/>	<input type="checkbox"/>	_____
Soil classified to USCS	<input type="checkbox"/>	<input type="checkbox"/>	_____
Soil samples sealed appropriately	<input type="checkbox"/>	<input type="checkbox"/>	_____
Soil samples stored appropriately (cooler, ice, etc.)	<input type="checkbox"/>	<input type="checkbox"/>	_____
Depth to water (if applicable)	<input type="checkbox"/>	<input type="checkbox"/>	_____
QC samples collected	<input type="checkbox"/>	<input type="checkbox"/>	_____
Samples collected in correct containers	<input type="checkbox"/>	<input type="checkbox"/>	_____
Water samples collected in correct order by analysis	<input type="checkbox"/>	<input type="checkbox"/>	_____
VOC samples (in VOAs) free of bubbles	<input type="checkbox"/>	<input type="checkbox"/>	_____
IDW stored and labeled appropriately	<input type="checkbox"/>	<input type="checkbox"/>	_____

**ADDITIONAL NOTES/COMMENTS:**  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

QA/QC Reviewer: \_\_\_\_\_

Signature: \_\_\_\_\_

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### SAP Worksheet #33—QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
DQA <ul style="list-style-type: none"> <li>Provides an overview of sampling, decontamination, and data storage procedures</li> <li>Identifies QC samples and summarizes associated analytical results</li> <li>Summarizes the findings of the analytical data validation process</li> <li>Provides an evaluation of data quality in accordance with the DQIs defined in the SAP in the final report</li> </ul>	Once	Approximately 60 days after completion of field investigation	Program QAM, KCH Project Chemist, KCH	Navy RPM
Laboratory System Audit Reports	During DoD ELAP assessment or renewal of DoD ELAP certification	TBD by DoD ELAP if offsite lab audit/ recertification is required.	DoD ELAP Laboratory Evaluator	DoD ELAP POC DoD ELAP EMAX, APPL Laboratory QAM
Field Sampling TSA Report	Once; audit will be conducted during sampling of soil and groundwater	Approximately 30 days after completion of audit	Program QAM, KCH	TOM, KCH Navy RPM
Report	Once	60 days after validated data received	TOM, KCH	Persons listed on Worksheet #3

Note:

DQA = data quality assessment

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### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Planning Documents	Evidence of approval and completeness of UFP-QAPP.	TOM, KCH	I	Internal
Field Notebooks	Field notebooks will be reviewed internally and placed into the project file for archival at project closeout.	Field Manager, KCH	I	Internal
COC Records and Shipping Forms	COC records and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the COC record will be initialed by the reviewer, a copy of the COC record retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	Field Manager, KCH	I	Internal
Sample Condition upon Receipt	Instances of discrepancies or missing or broken containers will be communicated to the Project Chemist or designee in the form of laboratory logins.	Laboratory PMs, EMAX and APPL Project Chemist, KCH	I	Internal and External
Sample Chronology	Holding times from collection to extraction or analysis and from extraction to analysis will be considered by the data validator during the data validation process.	Project Chemist, KCH Data Validator, LDC	I	Internal and External
EDDs	EDDs (100 percent) will be compared against hard-copy laboratory results.	Data Manager, KCH Data Validator, LDC	I	Internal and External
Analytical Laboratory Data	Laboratory data packages and EDDs will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. Received data packages and EDDs will be verified externally according to the SAP-specified analytical data validation procedures.	Project Chemist, KCH Laboratory PMs, EMAX and APPL Data Validator, LDC	I	Internal and External
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the site file. If CAs are required, a copy of the documented CA taken will be attached to the appropriate audit report in the QA site file. Periodically, and at the completion of site work, site file audit reports and CA forms will be reviewed internally to ensure that all appropriate CAs have been taken and that CA reports are attached. If CAs have not been taken, the site manager will be notified to ensure action is taken.	TOM, KCH Program QAM, KCH	I	Internal
CA Reports	CA reports will be reviewed by the Project Chemist or PM and placed into the project file for archival at project closeout.	Project Chemist, KCH Program QAM, KCH	I	Internal

### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Associated (batch or periodic) PT Sample Results	Evaluate the PT sample results against performance requirements as specified by the method, contract, or procedure.	Data Validator, TBD Program QAM, KCH	IIa and IIb	Internal and External
Communication Logs	Establish that the proper communication procedures were implemented by field and laboratory personnel.	PQAO, KCH	IIa	Internal
Copies of Laboratory Notebook, Records and Prep Sheets	Establish that the proper documentation was implemented by laboratory personnel.	Data Validator, LDC	IIa	External
CA Reports	Establish that the proper reporting procedures were implemented from laboratory personnel to laboratory QAM.	Data Validator, LDC Program QAM, KCH	IIa	Internal and External
Definitions of Laboratory Qualifiers	Assess that the laboratory data qualifiers were defined and properly assigned per the method, contract, or procedure.	Data Validator, LDC	IIa and IIb	External
Documentation of CA Results	Establish that the CA procedures were implemented and the CA properly addressed by laboratory QAM.	Data Validator, LDC Program QAM, KCH	IIa and IIb	External
Documentation of Individual QC Results (for example, spike, duplicate, LCS)	Establish that the QC results were properly reported and whether project performance criteria were met.	Data Validator, LDC	IIa and IIb	External
Documentation of Laboratory Method Deviations	Evaluate whether deviations from laboratory methods affected data and whether laboratory data qualifiers were assigned, if applicable.	Data Validator, LDC	IIa and IIb	External
EDDs	Assess whether required analytical data/values were provided by the laboratory in the proper EDD format.	Data Manager, KCH Project Chemist, KCH	IIa and IIb	Internal
Instrument Calibration Reports	Establish that instrument initial and continuing calibration were performed per the method, contract, or procedure, and deviations were documented.	Data Validator, LDC	IIa and IIb	External
Laboratory Name	Establish that analytical laboratories performing analysis are identified in analytical data reports.	Project Chemist, KCH	IIa	Internal
Laboratory Sample ID Numbers	Establish that unique laboratory sample ID numbers are used and are traceable to each unique sample ID, including QC samples.	Data Validator, LDC	IIa	External

### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
QC Sample Raw Data	Establish that QC samples (blanks, MS/MSD, LCS, surrogates, ISSs, etc.) were analyzed in accordance with the method and met the performance criteria.	Data Validator, LDC	IIa and IIb	External
QC Summary Information	Evaluate whether QC results met project performance criteria, verify that deviations were documented, and assess blank contamination in accordance with USEPA National Functional Guidelines (USEPA, 2011, 2014a, 2014b). Document in data validation checklists and validation reports.	Data Validator, LDC	IIa and IIb	External
Raw Data	Establish that sample preparation and analytical raw data (i.e., calculations, deviations, etc.) are correct and complete.	Data Validator, LDC	IIa and IIb	External
Reporting Forms Completed with Actual Results	Assess whether accurate and complete transcription of analytical data (i.e., analytical instrument output to reporting form) occurred and that QLs were achieved.	Data Validator, LDC	IIa and IIb	External
Signatures for Laboratory Sign-Off (for example, laboratory QAM)	Establish that each analytical data report was reviewed and signed by the laboratory QAM.	Data Validator, LDC	IIa	External
Standards Traceability Records	Establish that standards and reagents used during sample preparation and analysis are traceable and meet method, contract, and procedural requirements.	Program QAM, KCH	IIa	Internal
COC Records	Establish that the proper sample custody procedures were implemented by field personnel.	Project Chemist, KCH	IIa	Internal
Communication Logs	Establish that the proper communication procedures were implemented by field and laboratory personnel.	Program QAM, KCH PQAO, KCH Field Manager, KCH Project Chemist, KCH	IIa	Internal
CA Reports	Establish that the proper reporting procedures were implemented from field personnel to Program QAM.	Program QAM, KCH	IIa and IIb	Internal
Documentation of CA Results	Establish that the proper reporting procedures were implemented by Program QAM.	Program QAM, KCH	IIa and IIb	Internal
Documentation of Deviation from Methods	Evaluate whether deviations from sampling and field methods affected data.	Program QAM, KCH PQAO, KCH	IIa and IIb	Internal

### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Documentation of Internal QA Review	Establish that field and sampling procedures were implemented in accordance with the method, contract, or procedure, and deviations were documented.	PQAO, KCH	IIa and IIb	Internal
EDDs	Assess whether required field data/values have been provided in the proper EDD format.	Project Chemist, KCH Data Manager, KCH	IIa	Internal
Identification of QC Samples	Establish that QC samples were collected in accordance with the method, contract, or procedure.	Project Chemist, KCH	IIa and IIb	Internal
Sampling Instrument Decontamination Records	Establish that proper decontamination procedures were implemented by field sampling personnel.	Field Manager, KCH	IIa and IIb	Internal
Field Instrument Calibration Logs, If Applicable	Establish that field instrumentation requiring calibration was calibrated in accordance with the method, manufacturer's manual, or procedure.	Field Manager, KCH	IIa	Internal
Sampling Location and Plan	Establish that sample collection was performed at required sampling locations in accordance with the sampling plan.	TOM, KCH	IIa and IIb	Internal
Sampling Notes	Evaluate whether sampling information was recorded correctly and completely on sampling forms and that deviations were documented.	Field Manager, KCH	IIa and IIb	Internal
Sampling Report (from field team leader to PM describing sampling activities)	Evaluate whether deviations occurred and their potential impact to data.	Field Manager, KCH	IIa and IIb	Internal
External Audit Report	Review laboratory audit reports and accreditation and certification records for the laboratory's performance on specific methods.	Program QAM, KCH Project Chemist, KCH	IIa and IIb	Internal
External PT Sample Results	Evaluate the PT sample results against performance requirements as specified by the method, contract, or procedure.	Program QAM, KCH Project Chemist, KCH	IIa	Internal
Laboratory Assessment	Establish that the laboratory is in compliance with the current QA manual, accreditation and certification requirements, and regulatory requirements.	Program QAM, KCH Project Chemist, KCH	IIa	Internal
Laboratory QA Plan	Establish that the laboratory has a current QA manual that has been prepared in accordance with regulatory requirements.	Program QAM, KCH Project Chemist, KCH	IIa	Internal

### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Method Detection Limit Study Information	Establish that the laboratory has performed method detection limit studies on each instrument annually or in accordance with the method, contract, or procedure.	Program QAM, KCH Project Chemist, KCH	IIa and IIb	Internal
ELAP, NELAP, and DoD ELAP Accreditations	Establish that the laboratory has current ELAP, NELAP, and DoD ELAP accreditations for the analyses to be performed.	Program QAM, KCH Project Chemist, KCH	IIa	Internal
Data Validation of VOCs in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of SVOCs in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of PAHs in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of TPH-p in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External

### SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Data Validation of TPH-e in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of OCPs in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of PCBs in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Superfund Organic Data Review</i> (USEPA, 2014a)	Data Validator, LDC	IIa and IIb	External
Data Validation of Metals (Total and Dissolved) in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Inorganic Data Review</i> (USEPA, 2014b).	Data Validator, LDC	IIa and IIb	External
Data Validation of Hexavalent Chromium in Soil, Water	NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.  Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>National Functional Guidelines for Inorganic Data Review</i> (USEPA, 2014b).	Data Validator, LDC	IIa and IIb	External

**SAP Worksheets #34, #35, and #36: Data Verification and Validation (Steps I and IIa/IIb) Process Table**

Data Review Input	Description	Responsible for Verification (title, organization)	Step I /IIa <sup>a</sup> /IIb <sup>b</sup>	Internal/ External
Data Validation of Dioxins/Furans in Soil	<p>NAVFAC Southwest EWI No. 1. All data (except waste disposal data) will be validated at the following level: 10 percent at Level IV and 90 percent at Level III.</p> <p>Analytical methods and laboratory SOPs, as presented in this SAP, DoD QSM – Version 5.0 Final (2013), and <i>Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review</i> (USEPA, 2011).</p>	Data Validator, LDC	IIa and IIb	External

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## SAP Worksheet #37—Usability Assessment

The usability assessment process will evaluate and document the usability of the data by considering the project PARCCS parameters. The usability assessment will also assess whether the data will be suitable for the intended needs of the project. Every data type (e.g., sampling, field screening data, and laboratory analytical data) will be relevant to the usability assessment. Data usability will include the entry of analytical data validation flags applied by the third-party analytical data validation subcontractor to the project data, as well as an overall assessment of the analytical data and field QC samples.

The assessment will consider each type of data, the relationship to the entire data set, and the adequacy of the data to fulfill the project DQOs. The SDGs will be assessed for correctness, completeness, and compliance with method or project-specific QA/QC requirements, including the results of the independent analytical data validation process and contractual requirements. Analytical data validation will evaluate the data based on the PARCCS parameters defined in this SAP, and other method-specific performance requirements. The overall assessment process will also evaluate data usability based on the intended use of the data.

The intent of the DQA process will be to establish the PARCCS criteria and usability of the final results with respect to the project DQOs. Upon completion of analytical data validation, each data point will be assessed as not qualified, qualified as estimated (“J” or “UJ” qualified), or qualified as rejected (“R” qualified) based on the acceptance criteria, and analytical data validation flags will be added to the project data. These parameters will be based on the analytical data quality and will encompass the DQIs (i.e., PARCCS parameters) established in this SAP. Qualification will be given according to each sample’s SDG and will be based on the SAP and applicable laboratory and data validation SOPs. Analytical and contractual compliance and completeness levels will be assessed for each analytical parameter. Finally, the overall usefulness of the data will be established as related to the project DQOs.

### Data Quality Indicators

Quantifiable criteria, known as measurement performance criteria (or PARCCS parameters), are presented in this SAP. The PARCCS criteria will be the qualitative and quantitative indicators of data quality, and are defined in the following discussion.

### Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision will be measured by using laboratory duplicates and field duplicate samples. It will be expressed in terms of the RPD as follows:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:

RPD = relative percent difference

C<sub>1</sub> = concentration of sample or MS

C<sub>2</sub> = concentration of duplicate or MSD

## Accuracy

Accuracy is the degree of agreement of an observed measurement (or an average of the same measurement type) with an accepted reference or true value. Accuracy of analytical determinations will be measured using laboratory QC analyses, such as MS, MSD, LCS, and surrogate spike recoveries. Accuracy will be measured by evaluating the actual result against the known concentration added to a spiked sample, and will be expressed as %R as shown below:

$$\%R = \frac{S - U}{C_{sa}} \times 100$$

Where:

- %R = percent recovery
- S = Measured concentration of spiked aliquot
- U = Measured concentration of unspiked aliquot
- C<sub>sa</sub> = Concentration of spike added

## Representativeness

Representativeness is the reliability with which a measurement or measurement system reflects the true conditions under investigation. Representativeness is influenced by the number and location of the sampling points, sampling timing and frequency of monitoring efforts, and the field and laboratory procedures. The representativeness of data will be maintained by the use of established field and laboratory procedures and their consistent application.

## Comparability

Comparability expresses the confidence with which one data set can be compared to another based on USEPA-defined procedures, where available. If USEPA procedures are not available, the procedures have been defined or referenced in this SAP.

The comparability of data will be established through well-documented methods and procedures, SOPs, standard reference materials, QC samples, performance-evaluation study results, and by reporting each data type in consistent units.

## Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Analytical data validation and DQA will evaluate which data will be valid and which data will be rejected. Percent completeness will be defined as follows:

$$\text{Percent Completeness} = \frac{V}{T} \times 100$$

Where:

- V = Number of valid (not rejected) measurements over a given time
- T = Total number of planned measurements

The completeness goal for this project will be 90 percent for valid, usable data. If the completeness goal of the project is not achieved, a discussion regarding the limitations on the use of the project data will be included in the Usability Assessment section of the DQA report, which will be an appendix to the RI report.

## **Sensitivity**

Sensitivity is the measure of a concentration at which an analytical method can positively identify and report analytical results. The sensitivity of an analytical method will be indicated by the project-required LOD, LOQ, and DL values, as compared to the PALs.

## **Detection and Quantitation Limits**

The DL is the minimum concentration of an analyte that can be demonstrated to be different from zero, or a blank concentration with 99 percent confidence from background noise for a specific analytical method (i.e., 1 percent chance of false positive). The LOD is the minimum concentration of an analyte that can be demonstrated to be present with 99 percent confidence from background noise (i.e., 1 percent chance of false negative). The LOQ represents the lowest concentration of an analyte that can be quantified within specified limits of precision and accuracy during routine laboratory operating conditions in a sample matrix. The LOQs are contractually specified minimum QLs for specific analytical methods and sample matrices, such as soil or water, and will typically be several times the DL to allow for sample matrices.

Selected analytical methods and associated LOQs are typically capable of quantifying contaminants of concern at concentrations below the most stringent screening criteria. The LOQs will reflect the maximum sensitivity of current, routinely used analytical methods.

For this project, samples will be reported as estimated values (“J” qualified) if the concentrations are less than the LOQs but greater than the DLs or LODs.

## **Evaluative Procedures to Assess Overall Measurement Error**

The usability assessment process for the project will consist of reviewing the analytical data validation reports for usable analytical data (i.e., no validation qualifications or estimated “J”/“UJ” qualifications) and invalid or rejected (“R” qualified) analytical data, as well as evaluating the field and analytical data for discrepancies or deviations. This assessment will evaluate the impact of the discrepancies or deviations on the usability of the data and assesses whether the necessary information has been provided for use in the decision-making process. The assessment will evaluate whether there were deviations in sampling activities (e.g., incorrect sample location, improper or malfunctioning sampling equipment, or incorrect analysis performed), COC documentation, or holding times; compromised samples (i.e., damaged or lost samples) and the need to resample; or changes to SOPs or methods that could potentially affect data quality.

An evaluation of QC sample results will be performed to assess whether unacceptable QC results (e.g., blank contamination) affect data usability.

Other parameters to be evaluated during the usability assessment may include the following:

- Matrix effects – matrix conditions that might have affected the performance of the extraction or analytical method

- Site conditions – unusual weather conditions or site conditions that might have affected the sampling plan
- Identifying critical and noncritical samples or target analytes
- Background or historical data
- Data restrictions – data that do not meet the project DQOs or were “R” qualified might be restricted, but usable, as qualitative values for limited decision-making purposes

The data will be evaluated for overall PARCCS criteria for each matrix, analytical group, and concentration level. Data use limitations will be discussed in the DQA report for data that do not meet the project DQOs or DQIs.

### **Personnel Responsible for Data Usability Assessment**

Karin Kaiser, Project Chemist, KCH

Data Validation Subcontractor, LDC

### **Documentation of the Data Usability Assessment**

Usability assessment results will be reported in the DQA report as an appendix to the RI report.

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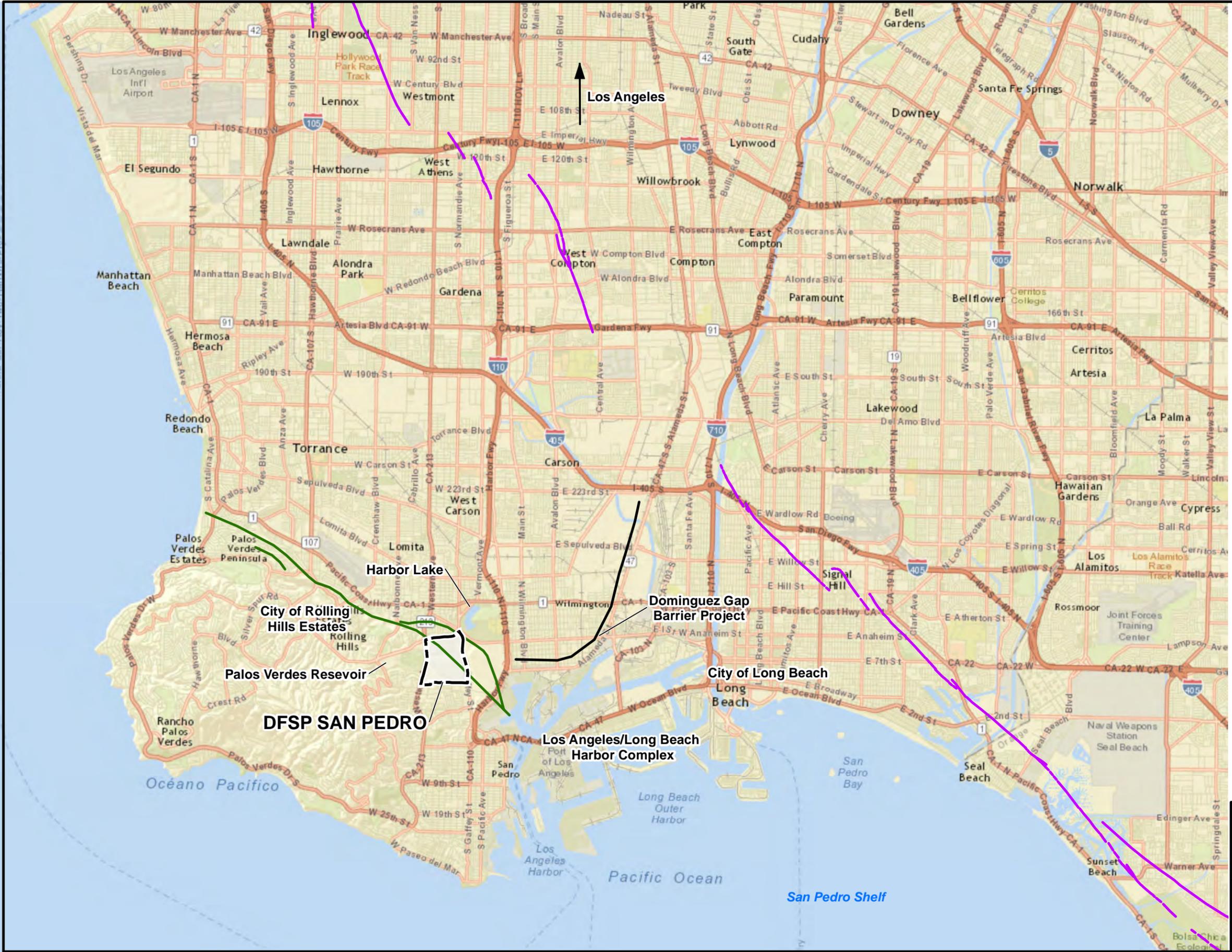
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## Figures

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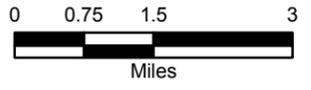
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- LEGEND**
-  NEWPORT-INGLEWOOD FAULT
  -  PALOS VERDES FAULT
  -  DFSP SAN PEDRO BOUNDARY

**NOTES:**  
 DFSP = Defense Fuel Support Point  
 IRP = Installation Restoration Program

**IMAGERY SOURCE:**  
 ESRI ArcGIS Online Web Service, Streets



**Site Vicinity**

Sampling and Analysis Plan, Remedial Investigation at  
 IRP Site 6, Defense Fuel Support Point San Pedro, California



N



FIGURE

**10-1**

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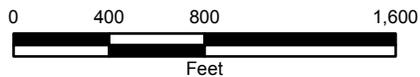
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**LEGEND**

-  IRP SITE BOUNDARY
-  FORMER NAVY HOUSING UNIT
-  DFSP SAN PEDRO BOUNDARY

NOTES:  
 - DFSP = Defense Fuel Support Point  
 - IRP = Installation Restoration Program



SOURCE:  
 ESRI ArcGIS Online Web Service,  
 World Imagery 5/5/2010

**IRP Site 6 Location**

Sampling and Analysis Plan, Remedial Investigation at  
 IRP Site 6, Defense Fuel Support Point San Pedro, California



FIGURE

**10-2**

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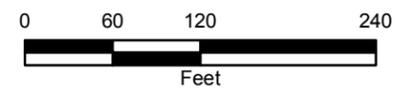
**LEGEND**

- EXISTING GROUNDWATER MONITORING WELL
- HISTORICAL SOIL BORING
- PROPOSED HAND AUGER BORING
- PROPOSED SOIL BORING
- PROPOSED GROUNDWATER MONITORING WELL
- PROPOSED TRENCH
- IRP SITE 6 BOUNDARY

NOTE:  
 - IRP = Installation Restoration Program

Approximate groundwater flow direction based on October 2013 groundwater elevation measurements (SGI, 2014).

IMAGERY SOURCE:  
 ESRI ArcGIS Online Web Service, World Imagery 5/25/2010



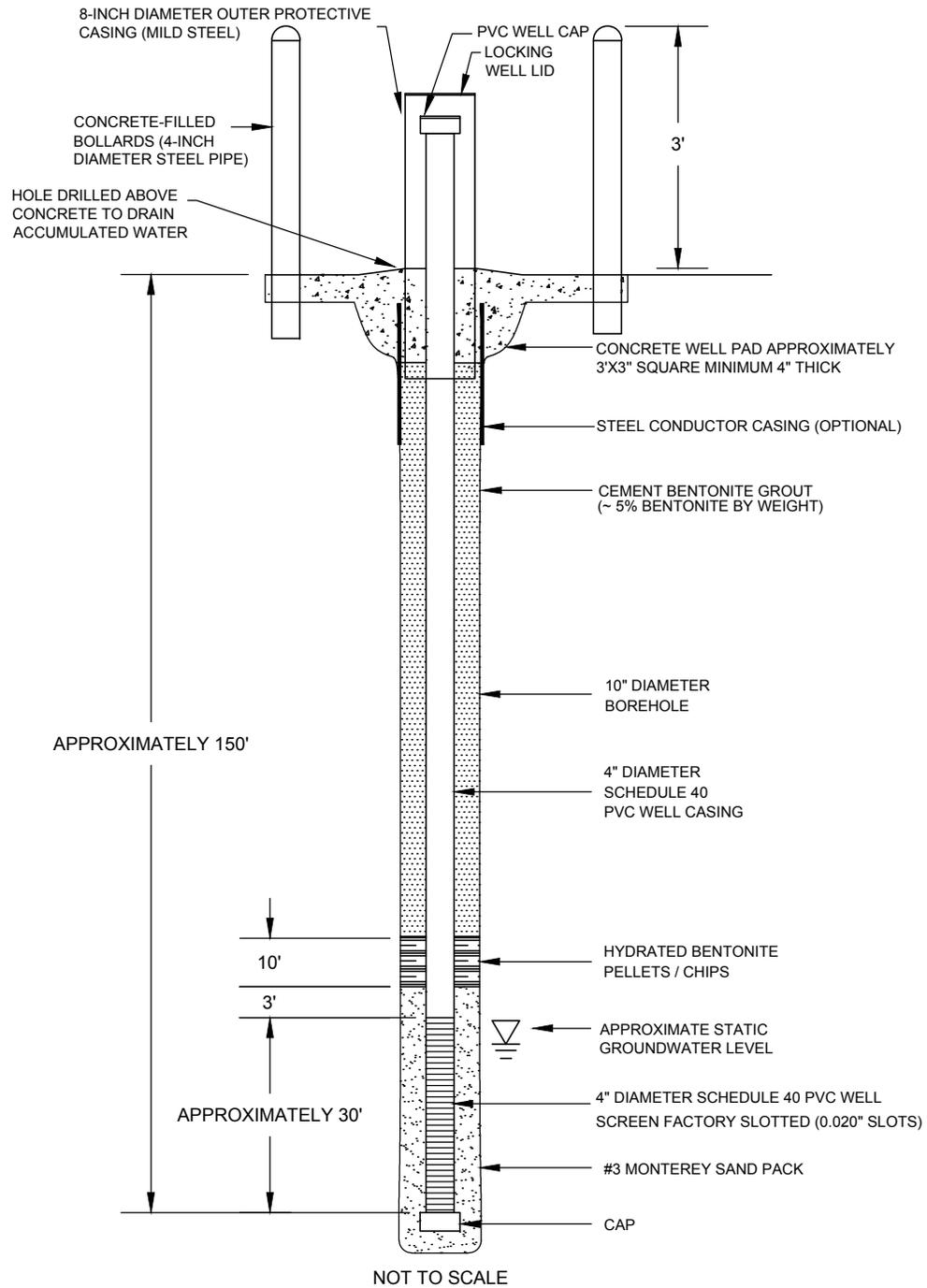
**IRP Site 6 Sample Locations**

Sampling and Analysis Plan, Remedial Investigation at  
 IRP Site 6, Defense Fuel Support Point San Pedro, California

N

FIGURE  
**10-3**

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NOTES:  
 PVC = Polyvinyl Chloride with threaded joints, no glued joints  
 IRP = Installation Restoration Program

<b>Proposed Groundwater Monitoring Well Construction Diagram</b>		
Sampling and Analysis Plan, Remedial Investigation at IRP Site 6, Defense Fuel Support San Pedro, California		
		FIGURE <b>14-1</b>

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**Attachment 1**  
**Site Photos**

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**Photo A1-1:** Southern part of IRP Site 6, looking north, with concrete v-drain (Source: Site walk, April 2014).



**Photo A1-2:** Southern part of IRP Site 6, looking northwest (Source: Site Walk, April 2014).



**Photo A1-3:** Northwest Branch at IRP Site 6, looking southeast (Source: Site Walk, April 2014).



**Photo A1-4:** Debris within Northeast Branch at IRP Site 6, looking north (Source: Site Walk, April 2014).



**Photo A1-5:** Debris within Northeast Branch at IRP Site 6, looking north (Source: Site Walk, April 2014).

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**Attachment 2**  
**Historical Soil Boring Locations and Analytical Data (Jacobs, 1993)**

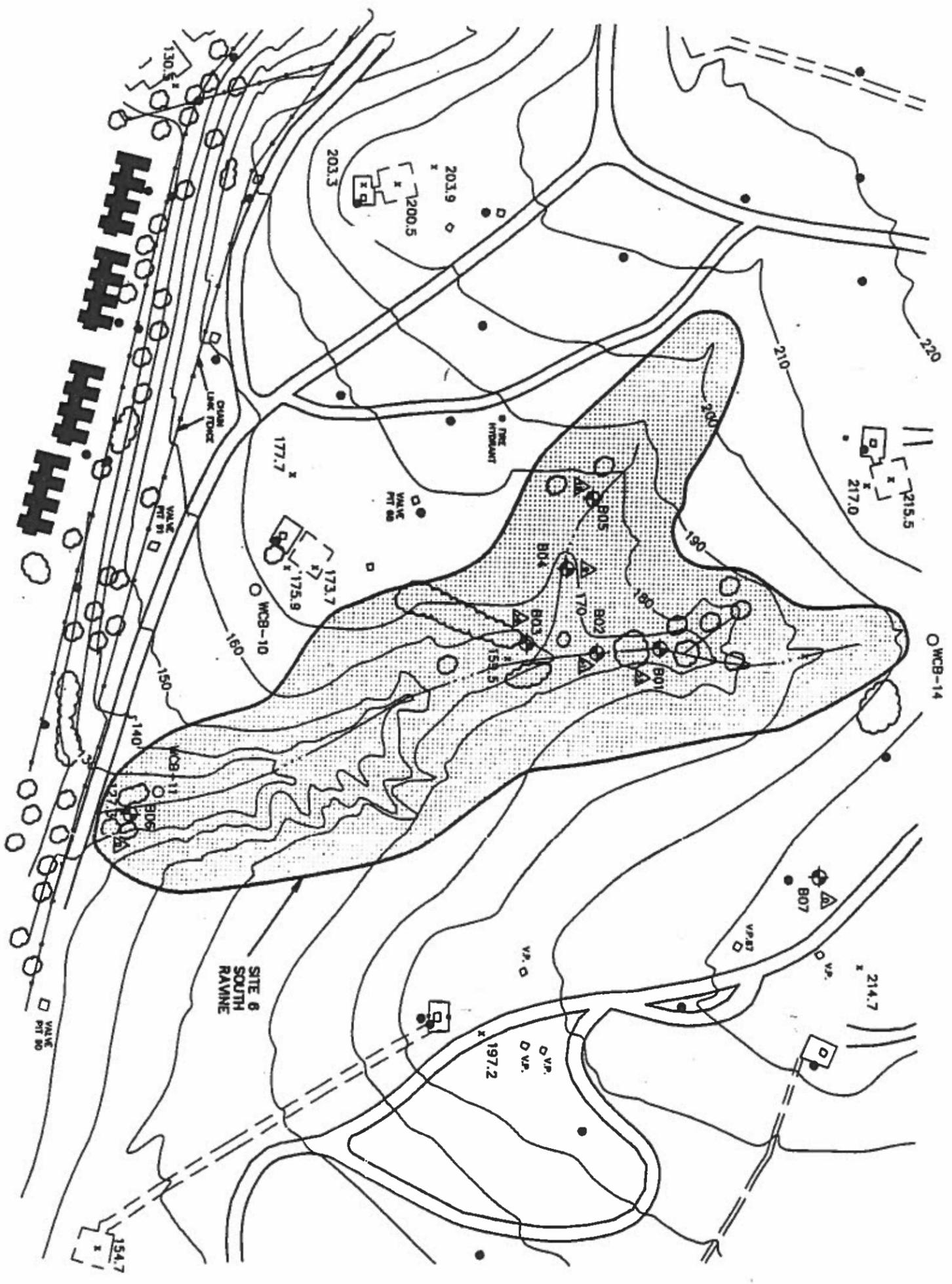
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TABLE 11.1
Site 6 - South Ravine
Soil Sample Analytical Results

Table with columns for Sample Number, Borehole, Depth, Compound, and various analytical results (e.g., Volatile Organics, Semivolatile Organics, Pesticides/PCBs, Diesel, Organic Lead, Metals). Rows include compounds like Methylene Chloride, Acetone, Benzene, and various metals like Aluminum, Iron, and Lead.

(1) All sample numbers are preceded by the prefix "B02A" in the laboratory data reports.
(2) Total TICs = the sum of all compounds tentatively identified by the method.
ND: analyte not detected
Qualifiers: Organic: J - estimated value; B - analyte found in blank as well as sample; N - presumptive evidence of compound present
Inorganics: J - estimated value; B - concentration exceeds instrument detection limit (IDL), but is less than contract required quantitation limit (CROL)
Gasoline and Diesel: Z - unknown aliphatic hydrocarbons calculated to gasoline/diesel concentrations respectively
Heavy lines surrounding data indicates the concentration exceeds the background threshold limit

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SOURCE: ROGOWAY/BORKOVETZ ASSOCIATES  
 DRAINAGE AND SLOPE REPAIRS,  
 SHEETS C-4, C-5, 1984



LEGEND

- 200 CONTOUR LINE
- FENCE
- ROAD AND STREET
- BUILDING
- VEGETATION LINE
- APPROXIMATE CENTER OF RAVINE
- UTILITY POLE
- 445 SPOT ELEVATION
- B01 SOIL BORING, JACOBS, 1992
- WCB-10 SOIL BORING, WOODWARD-CLYDE, 1990
- APPROXIMATE DEPTH TO NATURAL SOIL (FEET)



SCALE IN FEET

PROJ. MGR. <b>J. PENALBA</b>	<b>JACOBS ENGINEERING GROUP INC.</b> <small>PRINCIPAL OFFICE</small> 6750 SAN PEDRO FACILITY SAN PEDRO, CALIFORNIA
PROJ. ENG. <b>J. NICHOLSON</b>	
DRAWN BY <b>C. RICCIO</b>	
CHECKED BY <b>F144202A</b>	
DATE <b>03/18/93</b>	PROJECT NO. <b>01-F144-SP</b>
	FIGURE <b>11.1</b>

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**Attachment 3**  
**KCH SOPs**

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# Standard Operating Procedure - 1

Title: **Soil Sampling**

Document Number: **SOP 1**

**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Soil Sampling

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## I. Purpose and Scope

The purpose of this procedure is to provide guidelines for obtaining samples of surface and subsurface soils using hand and drilling-rig mounted equipment. Surface samples are defined as soil samples collected from 0 to 6 inches below ground surface or the first 2 inches of soil below a surficial layer of vegetation.

## II. Equipment and Materials

- Stainless-steel trowel, shovel, scoop, coring device, hand auger, or other appropriate hand tool
- Stainless-steel, split-spoon samplers
- Thin-walled sampling tubes
- Drilling rig or soil-coring rig
- Stainless-steel pan or bowl
- Stainless-steel spoon
- Sample bottles
- Encore™ or equivalent sampler

## III. Procedures and Guidelines

Before sampling begins, equipment will be decontaminated using the procedures described in SOP *Decontamination of Personnel and Equipment*. The sampling point location is recorded in the field logbook. Debris and or vegetation should be cleared from the sampling location prior to sampling. If the site is paved, the paving material should be removed using a breakout bar, jackhammer, or concrete coring. These activities should be conducted using specialist subcontractors where necessary and sampling-related activities be covered by the project-specific health and safety plan/accident prevention plan.

Underground utility clearance activities shall be conducted for all subsurface sampling where buried utilities may be encountered and in all cases where drilling equipment will advance deeper than 5 feet below ground surface (see SOP *Locating and Clearing Underground Utilities*).

### A. Surface and Shallow Subsurface Sampling

A shovel, post-hole digger, hand auger, or other tool can be used to remove soil to a point just above the interval to be sampled. A decontaminated sampling tool will be used to collect the sample when the desired sampling depth has been reached. Soil for organic and inorganic analyses that require homogenizing or compositing shall be placed in a decontaminated stainless steel bowl and mixed with a stainless steel spoon (disposable equipment may also be used). Soil for volatile organic analysis is not mixed or composited but is placed directly into the appropriate sample bottles. A stainless-steel or dedicated wooden tongue depressor is used to transfer the sample from the bowl to the container.

Alternatively, soil samples for analysis of volatile organic compounds (VOC) and total petroleum hydrocarbons-purgeable (TPH-p) may need to be collected using an Encore™ or equivalent sampler following USEPA Method 5035.

The soils removed from the excavated hole should be visually described in the field logbook, including approximated depths.

## **B. Split-Spoon Sampling**

Using a drilling rig or direct push rig, a hole is advanced to the desired depth. For split-spoon sampling conducted using a drill rig, the samples are collected following ASTM D 1586, *Standard Penetration Test Method for Penetration Test and Split-Barrel Sampling of Soils*. The sampler is lowered into the hole and driven to a depth equal to the total length of the sampler; typically, this is 24 inches. The sampler is driven in 6-inch increments using a 140-pound weight ("hammer") dropped from a height of 30 inches. The number of hammer blows for each 6-inch interval is counted and recorded. To obtain enough volume of sample for subsequent laboratory analysis, use of a 3-inch ID sampler may be required. Blow counts obtained with a 3-inch ID spoon do not conform to ASTM D 1586 and are therefore not be used for geotechnical evaluations.

For split spoon sampling using direct push equipment the sampler is lowered to the required sampling depth and then driven a depth not to exceed the length of the sampler.

Soil samples to be submitted to an analytical laboratory for testing may be collected in an unlined split-spoon sampler and transferred to glass jars for shipment to the laboratory; alternatively, a split-spoon sampler lined with thin wall brass or stainless steel sleeves may be used to both collect and containerize the sample.

Soil sampling for VOCs and TPH-p may require the use of an Encore™ or equivalent sampler as specified by USEPA Method 5035. The Encore™ or equivalent samples are to be collected immediately upon recovery of the split spoon sampler (lined or unlined) following method specific protocols.

The following steps should be followed when using an unlined sampler. Once retrieved from the hole, the sampler is carefully split open. Care should be taken not to allow material in the sampler to fall out of the open end of the sampler. To collect the sample, the surface of the sample should be removed with a clean tool and disposed of. Samples collected for volatiles analysis should be placed directly into the sample containers from the desired depth in the split spoon. Material for samples for all other parameters should be removed to a decontaminated stainless steel tray. The sample for organic (with the exception of VOC and TPH-p) and inorganic analyses may be homogenized and /or composited in the field by placing the sample in a clean bowl and breaking the sample into small pieces and removing gravel. The homogenized sample should be placed in the sample containers. If sample volume requirements are not met by a single sample collection, additional sample volume may be obtained by collecting a sample from below the sample and compositing the sample for non-volatile parameters only.

For sampling using a lined split-spoon sampler, the following steps shall be followed:

- Place clean and decontaminated sampler sleeves in the sampler barrel then assemble the sampler by aligning both sides of the barrel and attaching the drive shoe and sampler head to the bottom and top of the sampling barrel.
- After driving the sample using either drilling or direct push equipment retrieve the sampler from the borehole and disassemble it.

- Inspect the soil sample at the ends of each sampling tube and log the soil type as specified in SOP Logging of Soil Borings and assess if the sampling tube has been filled, then select which tubes to submit for analysis.
- Prepare each sampling tube for storage and transportation by sealing the ends of each sampling tube with Teflon sheeting and tightly fitting plastic end caps. The end caps shall be held in place with silicone tape or other USEPA approved sealing tape; electrical or duct tape shall not be used.
- Complete and attach a sampling label the sampling tubes and include the sample depth based on the location in the sampling interval from which the sampling tube was taken in the logbook.
- Decontaminate the sampler before taking the next sample.

### **C. Thin-Walled Tube Sampling for Geotechnical Investigations**

Undisturbed samples may be collected for analysis for geotechnical parameters such as vertical hydraulic conductivity. These samples will be collected using thin-walled sampling tubes (sometimes called Shelby tubes) according to ASTM D 1587. Tubes will be 24 to 36 inches long and 3 to 4 inches in diameter, depending upon the quantity of sample required. Samples for chemical analysis normally are not collected from thin-walled tube samples.

Undisturbed samples will be obtained by smoothly pressing the sampling tube through the interval to be sampled using the weight of the drilling rig. Jerking the sample should be avoided. Once the sample is brought to the surface, the ends will be sealed with bees wax and then sealed with end caps and heavy tape. The sample designation, date and time of sampling, and the up direction will be noted on the sampling tube. The tube shall be kept upright as much as possible and will be protected from freezing, which could disrupt the undisturbed nature of the sample.

## **IV. Key Checks and Preventive Maintenance**

- Check that decontamination of equipment is thorough.
- Check that sample collection is swift to avoid loss of volatile organics during sampling.

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# Standard Operating Procedure - 5

Title: **Logging of Soil Borings**

Document Number: **SOP 5**

**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Logging of Soil Borings

---

## I. Purpose and Scope

This standard operating procedure (SOP) document provides guidance to obtain accurate and consistent descriptions of soil characteristics during soil-sampling operations and other geologic, geotechnical and/or hazardous waste investigations. The characterization is based on visual examination and manual tests, not on laboratory determinations.

## II. Equipment and Materials

- Indelible pens
- Tape measure or ruler
- Field logbook
- Spatula
- Squirt bottle with water
- Rock- or soil-color chart (e.g., Munsell)
- Grain-size chart
- Hand lens

## III. Procedures and Guidelines

This section covers several aspects of the soil characterization: instructions for completing the CH2M HILL Kleinfelder, A Joint Venture (KCH) soil boring log form (attached), field classification of soil, and standard penetration test procedures.

### A. Instructions for Completing Soil Boring Logs

Soil boring logs will be completed in the field logbooks or on separate soil boring log sheets. Information collected will be consistent with that required for a KCH soil boring log form (attached), or an equivalent form that supplies the same information.

The information collected in the field to perform the soil characterization is described below.

### B. Heading Information

**Boring/Well Number.** Enter the boring/well number. A numbering system should be chosen that does not conflict with information recorded for previous exploratory work done at the site. Number the sheets consecutively for each boring.

**Location.** If station, coordinates, mileposts, or similar project layout information is available, indicate the position of the boring relative to that system using modifiers such as *approximate* or *estimated* as appropriate.

**Elevation.** Enter elevation data if known at time of drilling. Elevation may be determined at the conclusion of field activities.

**Drilling Contractor.** Enter the name of the drilling company and the city and state where the company is based.

**Drilling Method and Equipment.** Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger). Information on the drilling equipment (e.g., CME 55, Mobile B61) should also be noted.

**Water Level and Date.** Enter the depth below ground surface to the apparent water level in the borehole. The information should be recorded as a comment. If free water is not encountered during drilling or cannot be detected because of the drilling method, this information should be noted. Record date and time of day (for tides, river stage) of each water level measurement.

**Date of Start and Finish.** Enter the dates the boring was begun and completed. Time of day should be added if several borings are performed on the same day.

**Logger.** Enter the first initial and full last name.

## C. Technical Data

**Depth Below Surface.** Use a depth scale that is appropriate for the sample spacing and for the complexity of subsurface conditions.

**Sample Interval.** Note the depth at the top and bottom of the sample interval.

**Sample Type and Number.** Enter the sample type and number. Sample type will indicate the type of sampler used to collect the sample (e.g., split spoon). Sample numbers are typically specified in the work plan/sampling and analysis plan.

**Sample Recovery.** Enter the length to the nearest 0.1-foot of soil sample recovered from the sampler or the percentage of sample recovered compared to the length of the sampler. There will often be some wash or caved material above the sample; do not include the wash material in the measurement.

**Standard Penetration Test Results.** In this column, enter the number of blows required for each 6 inches of sampler penetration and the "N" value, which is the sum of the blows in the middle two 6-inch penetration intervals (in 18-inch samplers, sum the blows required to drive the bottom two 6-inch penetration intervals). A typical standard penetration test involving successive blow counts of 2, 3, 4, and 5 is recorded as 2-3-4-5. The standard penetration test is terminated if the sampler encounters refusal. Refusal is a penetration of less than 6 inches with a blow count of 50. A partial penetration of 50 blows for 4 inches is recorded as 50/4 inches. Penetration by the weight of the slide hammer only is recorded as WOH for weight of hammer.

**Sample Collection.** Samples may be collected using a split spoon sampler or via direct push sampling equipment. A 2-inch-diameter split spoon sampler should be driven using a 140-pound hammer, and a 3-inch-diameter split spoon sampler should be driven using a 300-pound hammer. Blow count data may be collected for all split spoon sampling, but N values only apply for the standard penetration test, which specifies the 2-inch-diameter sampler with the 140-pound hammer.

**Soil Description.** The soil classification should follow the format described in the "Field Classification of Soil" subsection below.

**Comments.** Include all pertinent observations (changes in drilling fluid color, rod drops, drilling chatter, rod bounce as in driving on a cobble, damaged Shelby tubes, and equipment malfunctions). In addition, note if casing was used, the sizes and depths installed, and if drilling fluid was added or changed. You should instruct the driller to alert you to any significant changes in drilling (changes in material, occurrence of boulders, and

loss of drilling fluid). Such information should be attributed to the driller and recorded in this column.

Specific information might include the following:

- The date and the time drilling began and ended each day
- The depth and size of casing and the method of installation
- The date, time, and depth of water level measurements
- Depth of rod chatter
- Depth and percentage of drilling fluid loss
- Depth of hole caving or heaving
- Depth of change in material
- Health and safety monitoring data
- Drilling interval through a boulder

## D. Field Classification of Soil

This section presents the format for the field classification of soil. In general, the approach and format for classifying soils should conform to American Society of Testing and Materials (ASTM) D 2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)*.

The Unified Soil Classification System is based on numerical values of certain soil properties that are measured by laboratory tests. It is possible, however, to estimate these values in the field with reasonable accuracy using visual-manual procedures (ASTM D 2488). In addition, some elements of a complete soil description (such as the presence of cobbles or boulders, changes in strata, and the relative proportions of soil types in a bedded deposit) can be obtained only in the field.

Soil descriptions should be precise and comprehensive without being verbose. The correct overall impression of the soil should not be distorted by excessive emphasis on insignificant details. In general, similarities rather than differences between consecutive samples should be stressed.

Soil descriptions must be recorded for every soil sample collected. The format and order for soil descriptions should be as follows:

1. Soil name (synonymous with ASTM D 2488 Group Name) with appropriate modifiers
2. Group symbol in parentheses – for example, (SP)
3. Color, using Munsell color designation
4. Moisture content
5. Relative density or consistency
6. Soil structure, mineralogy, or other descriptors

This order follows, in general, the format described in ASTM D 2488.

## E. Soil Name

The basic name of a soil should be the ASTM D 2488 Group Name on the basis of visual estimates of gradation and plasticity.

Examples of acceptable soil names are illustrated by the following descriptions:

- A soil sample is visually estimated to contain 15 percent gravel, 55 percent sand, and 30 percent fines (passing No. 200 sieve). The fines are estimated as either low or highly

plastic silt. This visual classification is SILTY SAND WITH GRAVEL, with a group symbol of (SM).

- Another soil sample has the following visual estimate: 10 percent gravel, 30 percent sand, and 60 percent fines (passing the No. 200 sieve). The fines are estimated as low plastic silt. This visual classification is SANDY SILT. The gravel portion is not included in the soil name because the gravel portion was estimated as less than 15 percent. The group symbol is (ML).

The gradation of coarse-grained soil (more than 50 percent retained on No. 200 sieve) is included in the specific soil name in accordance with ASTM D 2488. There is no need to further document the gradation. However, the maximum size and angularity or roundness of gravel and sand-sized particles should be recorded. For fine-grained soil (50 percent or more passing the No. 200 sieve), the name is modified by the appropriate plasticity/elasticity term in accordance with ASTM D 2488.

Interlayered soil should each be described starting with the predominant type. An introductory name, such as “Interlayered Sand and Silt,” should be used. In addition, the relative proportion of each soil type should be indicated (see Table 1 for example).

Where helpful, the evaluation of plasticity/elasticity can be justified by describing results from any of the visual-manual procedures for identifying fine-grained soils, such as reaction to shaking, toughness of a soil thread, or dry strength as described in ASTM D 2488.

**TABLE 1**  
Example Soil Descriptions

---

Poorly Graded Sand (SP)
Fat Clay (CH)
Silt (ML)
Well-Graded Sand with Gravel (SW)
Poorly Graded Sand with Silt (SP-SM)
Organic Soil with Sand (OH)
Silty Gravel with Sand (GM)
Lean Clay (CL)
Silty Sand with Gravel (SM)
Sandy Elastic Silt (MH)
Lean Clay with Sand (CL)
Well-Graded Gravel with Silt (GW-GM)

---

## F. Group Symbol

The appropriate group symbol from ASTM D 2488 must be given after each soil name. The group symbol should be placed in parentheses to indicate that the classification has been estimated.

In accordance with ASTM D 2488, dual symbols (e.g., GP-GM or SW-SC) can be used to indicate that a soil is estimated to have about 10 percent fines. Borderline symbols (e.g., GM/SM or SW/SP) can be used to indicate that a soil sample has been identified as having properties that do not distinctly place the soil into a specific group. Generally, the group

name assigned to a soil with a borderline symbol should be the group name for the first symbol. The use of a borderline symbol should not be used indiscriminately. Every effort should be made to first place the soil into a single group.

## G. Color

The color of a soil must be given. The color description should be based on the Munsell system. The color name and the hue, value, and chroma should be given.

## H. Moisture Content

The degree of moisture present in a soil sample should be defined as dry, moist, or wet. Moisture content can be estimated from the criteria listed on Table 2.

**TABLE 2**  
Criteria for Describing Moisture Condition

Dry
Moist
Wet

## I. Relative Density or Consistency

Relative density of a coarse-grained (cohesionless) soil or consistency of a clay or silt may be estimated in the field based on N-values (ASTM D 1586, *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils*). If the presence of large gravel, disturbance of the sample, or nonstandard sample collection makes determination of the in situ relative density or consistency difficult, then this item should be left out of the description and explained in the Comments column of the soil boring log.

Relationships for determining relative density or consistency of soil samples are given in Tables 3 and 4. Although typically part of geotechnical investigations, determination of relative density or consistency based on N-values may not be required for nongeotechnical investigations.

**TABLE 3**  
Relative Density of Coarse-Grained Soil

Blows/Foot	
0 – 4	very loose
5 – 10	loose
11 – 30	medium dense
31 – 50	dense
50	very dense

Developed from Sowers, G. F. *Introductory Soil Mechanics and Foundations: Geotechnical Engineering*. MacMillan Publishing Co., New York, 4th edition. 621 pp., 1979.

**TABLE 4**  
Consistency of Fine-Grained Soil

Blows/Foot	Consistency	Pocket Penetrometer (TSF)	Torvane (TSF)	Field Test
< 2	Very soft	< 0.25	< 0.12	Easily penetrated several inches by fist
2 – 4	Soft	0.25 – 0.50	< 0.12 – 0.25	Easily penetrated several inches by thumb
5 – 8	Firm	0.50 – 1.0	0.25 – 0.5	Can be penetrated several inches by thumb with moderate effort
9 – 15	Stiff	1.0 – 2.0	0.5 – 1.0	Readily indented by thumb, but penetrated only with great effort
16 – 30	Very stiff	2.0 – 4.0	1.0 – 2.0	Readily indented by thumbnail
30	Hard	> 4.0	> 2.0	Indented with difficulty by thumbnail

Developed from Sowers, G. F. *Introductory Soil Mechanics and Foundations: Geotechnical Engineering*. MacMillan Publishing Co., New York, 4th edition. 621 pp., 1979.

## J. Soil Structure, Mineralogy, and Other Descriptors

Discontinuities and inclusions are important and should be described. Such features include joints or fissures, slickensides, bedding or laminations, veins, root holes, and wood debris.

Significant mineralogical information such as cementation, abundant mica, or unusual mineralogy should be described.

Other descriptors may include particle size range or percentages, particle angularity or shape, maximum particle size, hardness of large particles, plasticity of fines, dry strength, dilatancy, toughness, and staining, as well as other information such as organic debris, odor, or presence of free product.

## K. Equipment and Calibration

Before starting the testing, the equipment should be inspected for compliance with the requirements of ASTM D 1586 (if applicable). The split-barrel sampler should have an outer diameter (OD) of 2 to 3 inches and should have a split tube at least 18 inches long. The minimum size sampler rod allowed is “A” rod (1 5/8-inch OD). A stiffer rod, such as an “N” rod (2 5/8-inch OD), is required for depths greater than 50 feet. The drive weight assembly should consist of a 140-pound or 300-pound hammer weight, a drive head, and a hammer guide that permits a free fall of 30 inches.

## IV. Attachments

KCH soil boring log form, boring log legend, and a completed example.

## V. Key Checks and Preventive Maintenance

- Check entries to the soil boring log and field logbook in the field. Because the samples will be disposed of at the end of fieldwork, confirmation and corrections cannot be made later.
- Check that sample numbers and intervals are properly specified.
- Check that drilling and sampling equipment is decontaminated using the procedures defined in SOP *Decontamination of Personnel and Equipment*.

Date Started: \_\_\_\_\_ Date Completed: \_\_\_\_\_ Location: \_\_\_\_\_  
 Logged By: \_\_\_\_\_ Drilling method: \_\_\_\_\_  
 Total Depth: \_\_\_\_\_ Hammer Wt: \_\_\_\_\_  
 Latitude: \_\_\_\_\_ Surface Conditions: \_\_\_\_\_  
 Longitude: \_\_\_\_\_ Surface Elevation: \_\_\_\_\_

Depth (feet)	Sample Number	Sample Type	Blows/Foot	Recovery (%)	OVA (ppm) PID/FID	USCS	Description	Remarks	Well Construction
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									



**LOG OF BORING NO.**

PLATE

PROJECT NO.

# UNIFIED SOIL CLASSIFICATION SYSTEM

MAJOR DIVISIONS		LTR	ID	DESCRIPTION	MAJOR DIVISIONS	LTR	ID	DESCRIPTION
COARSE GRAINED SOILS	GRAVEL AND GRAVELLY		GW	Well-graded gravels or gravel with sand, little or no fines.	FINE GRAINED SOILS		ML	Inorganic silts and very fine sands, rock flour or clayey silts with slight plasticity.
			GP	Poorly-graded gravels or gravel with sand, little or no fines.			CL	Inorganic lean clays of low to medium plasticity, gravelly clays, sandy clays, silty clays.
			GM	Silty gravels, silty gravel with sand mixture.			OL	Organic silts and organic silt-clays of low plasticity.
			GC	Clayey gravels, clayey gravel with sand mixture.			MH	Inorganic elastic silts, micaceous or diatomaceous or silty soils.
	SAND AND SANDY		SW	Well-graded sands or gravelly sands, little or no fines.			CH	Inorganic fat clays (high plasticity).
			SP	Poorly-graded sands or gravelly sands, little or no fines.			OH	Organic clays of medium high to high plasticity.
			SM	Silty sand.			Pt	Peat and other highly organic soils.
			SC	Clayey sand.		HIGHLY ORGANIC SOILS		



**Geoprobe, Direct Push Sample**

**Large Bore Discrete Soil Sampler, 1.5 in. O.D., 1.12 in. I.D.**

**Modified California Sampler, 2.5 in. O.D., 2 in. I.D.**

**California Sampler, 3.0 in. dia.**

**Hand Augered**



**Blank casing**

**Screened casing**

**Cement grout**

**Bentonite**

**Sand pack or gravel pack**

**OVA** Organic Vapor Analyzer

**PID** Total organic vapors (parts per million) measured by a photo-ionization device

**FID** Total Organic vapors (parts per million) measured by a flame-ionization device

**NA** Not Applicable

**Sharp Contact (observed)**

**Inferred Contact (contact not observed)**

**Gradational Contact (observed)**

**Water level observed in boring**

**Stabilized water level**

**NFWE** No free water encountered

**Notes:** Blow counts represent the number of blows a 140-pound hammer falling 30 inches required to drive a sampler through the last 12 inches of an 18 inch penetration.

The lines separating strata on the logs represent approximate boundaries only. The actual transition may be gradual. No warranty is provided as to the continuity of soil strata between borings. Logs represent the soil section observed at the boring location on the date of drilling only.

References to plasticity of cohesive soils are based on qualitative field observations and not on quantitative field or laboratory tests. Qualitative soil plasticity is noted solely to aid in stratigraphic correlation and is not intended for geotechnical characterization of soils.



## BORING LOG LEGEND

CTO-003 - PARCEL C DATA GAP INVESTIGATION  
HUNTERS POINT NAVAL SHIPYARD  
SAN FRANCISCO, CALIFORNIA

PLATE

**1**

PROJECT NO. **CTO 003**

Date Started: 1/7/10 Date Completed: 1/7/10 Location: Southeast edge of Building 214  
 Logged By: N. Berner Drilling method: Macro Core  
 Total Depth: 10.0 ft Hammer Wt: None  
 Latitude: \_\_\_\_\_ Surface Conditions: Asphalt  
 Longitude: \_\_\_\_\_ Surface Elevation: Estimated 3m

Depth (feet)	Sample Number Time	Sample Type	Blows/Foot	Recovery (%)	OVA (ppm) PID	USCS	Description	Remarks
1	1002N015 Time:14:40			80			<b>SANDY CLAY with GRAVEL (CL)</b> - dark brown, moist, soft, medium plasticity, trace fine gravel subangular	
2							<b>CLAYEY SAND (SC)</b> - gray, brown mottling, moist, medium dense, fine grained sand, trace fine gravel	
3	1002N016 Time:14:43							
4								
5	1002N017 Time:14:45			90				
6	1002N018 Time:15:00						<b>CLAYEY SAND (SC)</b> - dark brown, moist, medium dense, fine to coarse grained sand, shell fragments	
7								
8							<b>SAND (SP)</b> - dark brown, moist, medium dense, coarse grained sand, shell fragments	
9	1002N019 Time:15:02							
10	1002N020 Time:15:04						<b>SANDY CLAY (CL)</b> - dark brown, moist, soft, coarse grained sand, medium plasticity	
11							<b>Boring terminated at approximately 10 feet below ground surface.</b>	
12								

L:\2010\10\PROJECTS\105688\03.01\CTO\_003.GPJ



PROJECT NO. **CTO 003**

**LOG OF BORING NO. 214-S-01**

CTO-003 - PARCEL C DATA GAP INVESTIGATION  
 HUNTERS POINT NAVAL SHIPYARD  
 SAN FRANCISCO, CALIFORNIA

PLATE

**12**

1/27/2010 9:22:18 AM

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## Standard Operating Procedure - 9

Title: **Calibration and Measurement with Field Instruments**

Document Number: **SOP 9**

**Contract No: N62473-09-D-2622**

**NAVFAC Southwest CLEAN IV**

Technical Review by:



Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:



Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Calibration and Measurement with Field Instruments

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## I. Purpose and Scope

Provide general guidelines and standard methodologies for the use, calibration, and maintenance of field instruments.

## II. Definitions

None.

## III. Procedure

Field instruments are used for the collection of field data and measurement of various conditions observed during site investigations. In general, take the following steps when using field instruments:

1. Before field use, remove the instrument from its container and assemble and clean it according to the manufacturer's instructions.
2. Before commencement of field activities each day, calibrate the instrument according to the manufacturer's instructions. If manufacturer's instructions do not require daily calibration, record such instructions in the field logbook. Record the instrument's identification (ID) and serial number, along with the calibration process/results, in the field logbook and/or calibration logbook.
3. Use the instrument to make the appropriate physical/chemical measurements and clean/decontaminate the instrument, if necessary, after each measurement.
4. If erroneous measurements are observed or if changes in environmental conditions warrant re-calibration, re-calibrate the instrument as specified by the manufacturer. Record the re-calibration information in the field logbook and/or the calibration logbook.
5. At the end of each day, clean and decontaminate the instrument before returning it to the storage location. Recharge instrument as necessary.
6. Perform factory maintenance and calibration at the intervals specified by the manufacturer. Have repairs, maintenance, and calibration performed by trained individuals according to the manufacturer's requirements. Record repairs, maintenance, and calibration in the field logbook or instrument calibration/maintenance logbook.

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## Standard Operating Procedure - 11

Title: **Decontamination of Personnel and Equipment**

Document Number: **SOP 11**

**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:



Douglas Gilkey  
Technical Reviewer

12/19/14  
Date

Quality Review by:



Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14  
Date

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# Decontamination of Personnel and Equipment

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## I. Purpose

To provide general guidelines for the decontamination of personnel, sampling equipment, and monitoring equipment used in potentially contaminated environments.

## II. Scope

This is a general description of decontamination procedures.

## III. Equipment and Materials

- Distilled or deionized water
- Potable water; must be from a municipal water supplier, otherwise an analysis must be run for appropriate volatile and semivolatile organic compounds and inorganic chemicals (e.g., Target Compound List and Target Analyte List chemicals)
- 2.5 percent (W/W) Liquinox<sup>®</sup> and water solution
- Large plastic pails, tubs, or buckets for Liquinox<sup>®</sup> and water; scrub brushes; squirt bottles for Liquinox<sup>®</sup> solution; and water, plastic bags, and plastic sheets
- DOT-approved 55-gallon drum for disposal of waste
- Phthalate-free gloves such as Nitrile
- Decontamination pad and steam cleaner/high-pressure cleaner for large equipment

## IV. Procedures and Guidelines

### A. Personnel Decontamination

1. To be performed after completion of tasks whenever potential for contamination exists, and upon leaving the exclusion zone.
2. Wash boots in Liquinox<sup>®</sup> solution, then rinse with water. If disposable latex booties are worn over boots in the work area, rinse with Liquinox<sup>®</sup> solution, remove, and discard into appropriate waste receptacle as identified in the site-specific waste management plan.
3. Wash outer gloves in Liquinox<sup>®</sup> solution, rinse, remove, and discard into appropriate waste receptacle as identified in the site-specific waste management plan.
4. Remove disposable coveralls (Tyveks) and discard into appropriate waste receptacle as identified in the site-specific waste management plan.
5. Remove respirator (if worn).
6. Remove inner gloves and discard into appropriate waste receptacle as identified in the site-specific waste management plan.
7. At the end of the work day, shower entire body, including hair, either at the work site or at home.
8. Sanitize respirator if worn.

**B. Sampling Equipment Decontamination—Groundwater Sampling Pumps**

Sampling pumps are decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Spread plastic sheeting on the ground to keep equipment from touching the ground.
3. Turn off pump after sampling. Remove pump from well and remove and dispose of tubing. Place pump in decontamination tube or clean bucket.
4. Turn pump back on and pump 1 gallon of Liquinox<sup>®</sup> solution through the sampling pump.
5. Rinse with 1 gallon of tap water.
6. Rinse with 1 gallon of distilled water.
7. Keep decontaminated pump in decontamination tube or remove and wrap in aluminum foil.
8. Collect all rinsate and dispose of in an appropriately labeled DOT-approved 55-gallon drum.
9. Decontamination materials (e.g., plastic sheeting, tubing) that have come in contact with used decontamination fluids or sampling equipment will be disposed of in an appropriate waste receptacle as identified in the site-specific waste management plan.

**C. Sampling Equipment Decontamination—Other Equipment**

Reusable sampling equipment is decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Rinse and scrub with potable water.
3. Wash all equipment surfaces that contacted the potentially contaminated soil/water with Liquinox<sup>®</sup> solution.
4. Rinse with potable water.
5. Rinse with distilled water.
6. Completely air dry and wrap exposed areas with aluminum foil (shiny side out) for transport and handling if equipment will not be used immediately.
7. Collect all rinsate water and dispose of in an appropriately labeled, DOT-approved 55-gallon drum.
8. Decontamination materials (e.g., plastic sheeting, tubing) that have come in contact with used decontamination fluids or sampling equipment will be disposed of as required by the project-specific waste management plan.

**D. Health and Safety Monitoring Equipment Decontamination**

1. Before use, wrap soil contact points in plastic to reduce need for subsequent cleaning.
2. Wipe all surfaces that had possible contact with contaminated materials with a paper towel wet with Liquinox<sup>®</sup> solution, and three times with a towel wet with distilled water. Dispose of all used paper as required by the project-specific waste management plan.

## **E. Sample Container Decontamination**

The outsides of sample bottles or containers filled in the field may need to be decontaminated before being packed for shipment or handled by personnel without hand protection. The procedure is:

1. Wipe container with a paper towel dampened with Liquinox<sup>®</sup> solution. Repeat the above step using potable water.
2. Dispose of all used paper towels as required by the project-specific waste management plan.

## **F. Heavy Equipment and Tools**

Heavy equipment such as drilling rigs, drilling rods/tools, and the backhoe will be decontaminated upon arrival at the site and between locations as follows:

1. Set up a decontamination pad in area designated by the Facility.
2. Steam clean heavy equipment until no visible signs of dirt are observed. This may require wire or stiff brushes to dislodge dirt from some areas.

## **V. Attachments**

None.

## **VI. Key Checks and Items**

Clean with solutions of Liquinox<sup>®</sup>, and distilled water.

- Do not use acetone or methanol for decontamination.
- Establish a waste management plan that clearly specifies which wastes are to be containerized and handled as hazardous waste, and which materials may be disposed of as regular trash.
- Decontaminate filled sample bottles before relinquishing them to anyone.

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# Standard Operating Procedure - 13

Title: **Preparing Field Logbooks**

Document Number: **SOP 13**

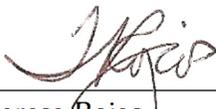
**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:

  
\_\_\_\_\_  
Douglas Gilkey  
Technical Reviewer

12/19/14  
Date

Quality Review by:

  
\_\_\_\_\_  
Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14  
Date

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# Preparing Field Logbooks

---

## I. Purpose

To provide general guidelines for entering field data into logbooks during site investigation and remediation field activities.

## II. Scope

This is a general description of data requirements and format for field logbooks. Logbooks are needed to properly document field activities in support of data evaluation and possible legal action. In addition to field logbooks, project-specific work plans may have additional requirements for documentation of measurements or other observations in the field.

## III. Equipment and Materials

- Logbook
- Indelible pen

## IV. Procedures and Guidelines

Properly completed field logbooks are a requirement of much of the work we perform under the Navy Comprehensive Long-Term Environmental Action (CLEAN) contract. Logbooks are legal documents and, as such, must be prepared following specific procedures and contain required information to ensure their integrity and legitimacy. This standard operating procedure (SOP) describes the basic requirements for field logbook entries.

### A. Procedures for Completing Field Logbooks

1. Field notes are commonly kept in bound logbooks. Pages should be water-resistant and notes should be taken only with waterproof, non-erasable permanent ink, such as that provided in Sanford Sharpie® permanent markers.
2. On the inside cover of the logbook the following information should be included:
  - Company name and address
  - Log-holder's name if logbook was assigned specifically to that person
  - Activity or location
  - Project name
  - Project manager's name
  - Phone numbers of the company, supervisors, and emergency response
3. All lines of all pages should be used to prevent later additions of text, which could later be questioned. Any line not used should be marked through with a line and initialed and dated. Any pages not used should be marked through with a line, the author's initials, the date, and the note "Intentionally Left Blank".
4. If errors are made in the logbook, cross a single line through the error and enter the correct information. All corrections shall be initialed and dated by the personnel performing the correction. If possible, all corrections should be made by the individual who made the error.
5. Daily entries will be made chronologically.
6. Information will be recorded directly in the field logbook during the work activity. Information will not be written on a separate sheet and then later transcribed into the logbook.

7. Each page of the logbook will have the date of the work and the note taker's initials.
8. The final page of each day's notes will include the note-taker's signature as well as the date.
9. Only information relevant to the subject project will be added to the logbook.
10. The field notes will be copied and the copies sent to the Project Manager or designee in a timely manner (at least by the end of each week of work being performed).

## **B. Information to be Included in Field Logbooks**

1. Entries into the logbook should be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. Entries must be legible and complete.
2. General project information will be recorded at the beginning of each field project. This will include the project title, the project number, and project staff.
3. Scope: Describe the general scope of work to be performed each day.
4. Weather: Record the weather conditions and any significant changes in the weather during the day.
5. Tail Gate Safety Meetings: Record time and location of meeting, who was present, topics discussed, issues/problems/concerns identified, corrective actions or adjustments made to address concerns/problems, and other pertinent information.
6. Standard Health and Safety Procedures: Record level of personal protection being used (e.g., level D PPE), record air-monitoring data on a regular basis, and note where data were recorded (e.g., reading in borehole, reading in breathing zone). Record other required health and safety procedures as specified in the project-specific health and safety plan.
7. Instrument Calibration: Record calibration information for each piece of health and safety and field measurement equipment. Include serial number of equipment calibrated, lot number and concentrations of calibration standards used, standard lot numbers, name of person who calibrated the equipment, the date/time calibrated, and whether it was within control limits. If not within control limits, record corrective actions taken.
8. Personnel: Record names of all personnel present during field activities and list their roles and their affiliation. Record when personnel and visitors enter and leave a project site and their level of personal protection.
9. Communications: Record communications with Project Manager, subcontractors, regulators, facility personnel, and others that impact performance of the project.
10. Time: Keep a running time log explaining field activities as they occur chronologically throughout the day.
11. Deviations from the Work Plan: Record any deviations from the work plan, why these were required, who approved the deviations, and any communications authorizing these deviations. Refer to any change management actions taken for the deviations.
12. Health and Safety Incidents: Record any health and safety incidents and immediately report any incidents to the Project Manager.
13. Subcontractor Information: Record name of company, names and roles of subcontractor personnel, types of equipment being used, and general scope of work. List times of starting and stopping work and quantities of consumable materials used if they are to be billed to the project.

14. **Problems and Corrective Actions:** Clearly describe any problems encountered during the field work and the corrective actions taken to address these problems. Document the personnel who put the corrective actions into place, who were notified of the problems, and who approved corrective actions taken.
15. **Technical and Project Information:** Describe the details of the work being performed. The technical information recorded will vary significantly between projects. The project work plan will describe the specific activities to be performed and may also list additional requirements for note taking and recording of measurements taken in the field. Discuss note-taking expectations with the Project Manager prior to beginning the field work. Also designate who will be check the field log books and frequency of the checks.
16. **Conditions:** Describe any conditions that might adversely affect the work or any data obtained (e.g., nearby construction that might have introduced excessive amounts of dust into the air). Describe any change in conditions that may affect the approach to the work.
17. **Sampling Information:** Specific information that will be relevant to most sampling jobs includes the following.
  - Description of the general sampling area – site name, buildings, and streets in the area
  - Station/location identifier
  - Description of the sample location – estimated location in comparison to two fixed points (Draw a diagram in the field logbook indicating sample location relative to these fixed points and include distances in feet.)
  - Description of the sample (sandy, clay, sheen, odor, etc.)
  - Names of samplers
  - Sample matrix and type
  - Sample date and time
  - Sample identifier
  - Draw a box around the sample ID so that it stands out in the field notes
  - Information on how the sample was collected (Distinguish between grab, composite, and discrete samples.) Describe the conditions and reasons for selecting the sample point.
  - Number and type of sample containers collected
  - Type of preservatives or preservation used
  - Any field measurements taken (i.e., pH, turbidity, dissolved oxygen, temperature, and conductivity)
  - Parameters to be analyzed for, if appropriate
  - Descriptions of soil samples and drilling cuttings (entered in depth sequence), along with Photoionization Detector readings and other observations (Include any unusual appearances of the samples.)
  - Chain-of-Custody Form number, if applicable
  - Shipping number/airbill number, if applicable
  - Quality Control samples taken and the parent samples for duplicates/replicates

### **C. Suggested Format for Recording Field Data**

1. Use the left-side border to record times and the remainder of the page to record information (see attached example).
2. Use tables to record sampling information and field data from multiple samples.

3. Sketch sampling locations and other pertinent information.
4. Sketch well-construction diagrams.

## V. Attachments

Example field notes.

67996

SPAWR

POINT LAND



*"Rite in the Rain"*

ALL-WEATHER

**LEVEL**

No. 311

619.553.4622 security emergency  
gate 503 inside gate A34 - SAN PEDRO POWER



"*Rite in the Rain*"  
ALL-WEATHER WRITING PAPER

Name KLENFELDEL

Address 5015 SHOREHAM PL  
SAN DIEGO, CA 92122

Phone 858.320.2000

Project SPAWN  
POINT LOMA

Clear Vinyl Protective Slipcovers (Item No. 30) are available for this style of notebook.  
Helps protect your notebook from wear & tear. Contact your dealer or the J. L. Darling Corporation

CONTENTS

PAGE	REFERENCE	DATE
2	NON-TIME SENSITIVE REMOVAL ACTION AT IEP SITES 9 AND 23, STANFORD POINT LOMA	10/2/07
5	" " " " " "	10/5/07
11	" " " " " "	10/11/07
15	" " " " " "	10/17/07
22	" " " " " "	10/18/07
30	" " " " " "	10/19/07
36	" " " " " "	10/22/07
44	" " " " " "	10/23/07

~~10/23/07~~

DATE OCTOBER 12, 2007

PROJECT NON-TIME CRITICAL Removal Action

AT IIP SITES 9 and 23

LOCATION - SPANAW, POINT LOMA, SANDHEDGE, CA

ACTIVITY - GEOPHYSICAL SURVEY, GPS LOCATING

CREW KUEHNFELDER - HOLLY CHAYKED (HC)

CDM - TONY GIMARAS (TG), JON BUSH (JB)

NAVFAC - ALLISON (RPM)

UHS - CHRIS

EQUIPMENT TRIMBLE GEO XT (KUEHNFELDER)  
geophysics cont. (UHS) - utility locate

WEATHER 60-70°F, sunny

0800 HC leaves for site

0825 HC onsite at bldg 27 (PASS & DEVAL),  
meet TG, JB.

0830 RPM on site

0845 UHS on site. All present into BAGE office

0900 Move to site, escorted by RPM.

- gate locked. RPM c/ site rep (CHRIS HOWARD)

0910 site rep c/ security.

- Security alert at another spanaw building,  
so all security personnel at that building  
right now.

0920 HC conduct large H&S meeting.

- all present sign SHASP.

HC

0930 HC/TG offsite for water while wait for  
security

0945 HC/TG onsite... still waiting for security.

1000 security on-site, open gates. Security  
officer advises gates MUST be locked behind  
crew. HC expresses safety concern. Security  
advises must be locked.

- HC req at least someone come out to

check on safety every 30 minutes or so.

- CHRIS HOWARD acknowledges safety concern,  
especially since little or no cell phone  
service.

1005 security stays for a while. HC/TG begin  
GPS utility excavation extent (overestimate)

locate and mark with orange pin and

pink tape where able to access.

- HC adv JB/TG unable to access some

locations (F and southeast of M)

- locations A, B, C, D lumped together

in one big area.

- locations J, K, L, M lumped together

in one big area, except unable to

get southeast of M.

- HC adv JB area will need to be brushed  
with accurate GPS or geophysics

HC

4 10/12/07 cont

- can be completed. JB ack.  
- main areas were "outlined" but geophysics unable to access internal areas due to THICK brush.

1140 He/Ta complete GPS as possible  
- Allison (rpm) and christina and security guard off site. He, Ta, JB, UCS locked inside gated area.

- JB of security to req someone unlock gate.

1150 security on site

1200 He/Ta off site

~~10/12/07~~

END

DATE 10/15/07

PROJECT IPT STOPS 9 a.m. - 23 removal action

LOCATION SPANNA POINT LOMA SAN DIEGO, CA

ACTIVITY BRUSHING, STOCKPILE SETUP

CREW KUENFELDER - HOLLY CARTER (HE)

BRAD ERSKINE (BE)

CDM - TONY GUNNS (TZ) JON BUSH (JB)

GEM - DAVID ARANDA (DA)

MARCEL MONTEPUSIO (MM)

MARIO TURADO (MT)

RON GEMTILE (RG)

EQUIPMENT TRIMBLE GEO XT (GPS)

BRUSHING -> EXCAVATOR USED (GEM)

0800 He leave for site. WINDY 60°F, cloudy

0830 He meets BE at bldg 27, move to site

0900 He/BE meet TG, JB, DA, MM, MT, RG

at gate outside of PPS sites 9 a.m. - 23.

JB adv issues with badges, so PPM-Allison

is obtaining visitor badges for everyone.

0905 He/BE/JB conduct H&S meeting.

He/BE adv concern vgd gate being

locked. JB adv gate will be unlocked

for entry. All present sign H&S.

0910 BE adv concern that all proposed

locations must be properly cleared for

He

6 10/15/07 - cont.

utilitics prior to beginning excavations.  
JBack and adv NAVY is sending utility  
crews and today or tomorrow  
MIS can come back out if necessary.  
JB adv all locations will be properly  
marked prior to excavation.  
BE adv.

0930 JB escorts crew to pass and demand  
to meet PPM with visitor badges.

1000 JB back with GEM & PPM Allison.  
PACIFIC ENERGY security on-site to unlock  
gate.

- HC/BE on-site, go to marked locations.  
1010 BE off-site.

1015 HC double checking GPS marked locations  
versus map locations (paper). GPS locations  
appear to be "off" approximately 20-30 feet

- HC / GREG TOOTH - KURT FERRELL GIS person  
to see if there is a coordinate system error  
or quick fix, but NA. HC changes GPS  
from NAD 83 to NAD 27, but no change.

HC changes to UTM, but no change HC /  
BE / M adv will NOT use GPS to mark  
locations but rather will use paper map  
which shows historic locations and

HC

10/15/07 cont

topography to more accurately  
mark locations.

1030 HC / TG begin by marking center  
point of proposed excavation "G", based  
on locating stakes for B42, B41, B43, B40  
(all historic on map). HC / TG adv GEM to  
clear brush in 20-foot radius from that

point.

1100 move to location "H", locate B66,  
B65, B70, B68 and B73, B72,  
fire hydrant - mark B90 as  
center of excavation "H".

HC adv GEM to remove brush  
in a 20-foot radius from that point

1130 move to location "I", locate  
B13, B94, B96, B95. mark  
historic location B11 as the  
centerpoint of excavation "I".

HC adv GEM to clear brush in a  
20-foot radius of that point.

1200 HC / TG begin to determine a  
"trapezoid" shaped area surrounding  
excavations "J", "K", "L", "M" as unable to  
access areas due to thick brush.

locate B10, B106 and mark SW corner as

HC

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# Standard Operating Procedure - 16

Title: **Chain-of-Custody**

Document Number: **SOP 16**

**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Chain-of-Custody

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## I. Purpose

The purpose of this standard operating procedure (SOP) is to provide information on sample chain-of-custody procedures to be used under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Program.

## II. Scope

This procedure describes the steps necessary for documenting the transfer or possession of samples through the use of Chain-of-Custody Forms. A Chain-of-Custody Form is required, without exception, for the tracking and recording of samples collected for onsite or offsite analysis (chemical or geotechnical) during program activities (except wellhead samples taken for measurement of field parameters). Use of the Chain-of-Custody Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis. This procedure identifies the necessary custody records and describes their completion. This procedure does not take precedence over region specific or site-specific requirements for chains of custody.

## III. Definitions

**Chain-of-Custody Form** - A printed, two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to another custodian. One copy of the form must be retained in the project file.

**Custodian** - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under one's custody if:

- It is in one's actual possession.
- It is in one's view, after being in one's physical possession.
- It was in one's physical possession and then he/she locked it up to prevent tampering.
- It is in a designated and identified secure area.

**Sample** - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

## IV. Responsibilities

**Project Manager** - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for development of documentation of procedures that deviate from those presented herein. The Project Manager is responsible for ensuring that chain-of-custody procedures are implemented. The Project Manager also is responsible for determining that custody procedures have been met by the analytical laboratory.

**Field Team Leader** - The Field Team Leader is responsible for determining that chain-of-custody procedures are implemented up to and including release to the shipper or laboratory. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to initiate chain-of-custody procedures, and maintain custody of samples until they are relinquished to another custodian, the sample shipper, or to a common carrier.

## V. Procedures

The term chain-of-custody refers to procedures that ensure evidence presented in a court of law is valid. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the laboratory and final place of storage or disposal, as well as providing security for the evidence as it is moved and/or passed from the custody of one individual to another.

Chain-of-custody procedures, recordkeeping, and documentation are an important part of the management and control of samples. Regulatory agencies must be able to provide the chain-of-possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

### A. Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When in situ measurements are made, the data are recorded directly in bound logbooks or other field data records with identifying information.

Information to be recorded in the field logbook when in situ measurements or samples for laboratory analysis are collected includes:

- Field sampler(s)
- Contract Task Order (CTO) number
- Project sample number
- Sample location or sampling station number
- Date and time of sample collection and/or measurement
- Field observations
- Equipment used to collect samples and measurements
- Calibration data for equipment used

Measurements and observations shall be recorded using waterproof ink.

### B. Sample Label

Samples, other than for in situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling and Analysis Plan. Each sample container is identified by a sample label (see Attachment A). Sample labels are provided, along with sample containers, by the analytical laboratory. The information recorded on the sample label includes:

- Project - Task Order (TO) name and number
- Station location - The unique sample number identifying this sample
- Date - A six-digit number indicating the day, month, and year of sample collection (e.g., 01/21/14)
- Time - A four-digit number indicating the 24-hour time of collection (e.g., 0954 is 9:54 a.m., and 1629 is 4:29 p.m.)
- Preservation – Type and quantity of preservation added.

- Analysis - VOA, BNAs, PCBs, pesticides, metals, cyanide, other
- Sampled By - Printed name of the sampler

Using only the work assignment number (as sample delivery group or work order) of the sample label maintains the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing the analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site). The field team should follow the sample ID system specified in the project-specific work plan and Sampling and Analysis Plan.

### C. Chain-of-Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

### D. Field Custody Procedures

- Samples are collected as described in the site Sampling and Analysis Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the Chain-of-Custody Form exactly.
- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
- A Chain-of-Custody Form will be prepared for each individual cooler shipped and will include **only** the samples contained within that particular cooler (or copies can be made of all Chain-of-Custody Forms and placed in all coolers). A Chain-of-Custody Form example is shown in Attachment B. The Chain-of-Custody Form for that cooler will then be sealed in a Ziploc bag and placed in the cooler prior to sealing. This ensures that the laboratory properly attributes trip blanks with the correct cooler and allows for easier tracking should a cooler become lost during transit.

### E. Transfer of Custody and Shipment

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Chain-of-Custody Form. This form documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Form is filled out as given below:

- Enter header information (TO number, samplers, and project name).
- Enter sample specific information (sample number, media, sample analysis required, and analytical method, sample preservation, sample type [composite, grab, wipe], number and type of sample containers, and date/time sample was collected).
- Sign, date, and enter the time under "Relinquished by" entry.
- Have the person receiving the sample sign the "Received by" entry. If shipping samples by a common carrier, print the carrier to be used in this space (i.e., Federal Express).
- If a carrier is used, enter the airbill number under *Remarks*, in the bottom right corner.
- Place the original (top, signed copy) of the Chain-of-Custody Form in a plastic zipper-type bag or other appropriate sample-shipping package. Retain the copy with field records.
- Sign and date the custody seal, a 1-inch by 3-inch white paper label with black lettering and an adhesive backing. Attachment C is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals shall be provided by the analytical laboratory.

- Place the seal across the shipping container opening (front and back) so that it would be broken if the container were to be opened.
- Complete other carrier-required shipping papers.

The custody record is completed using waterproof ink. Any corrections are made by drawing a single line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers will usually not accept responsibility for handling Chain-of-Custody Forms; this necessitates packing the record in the shipping container. As long as custody forms are sealed inside the shipping container and the custody seals are intact, commercial carriers are not required to sign the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Form, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

## VI. Quality Assurance Records

Once samples have been packaged and shipped, the Chain-of-Custody Form copy and airbill receipt become part of the quality assurance record.

## VII. Attachments

- A - Sample Label
- B - Chain of Custody Form
- C - Custody Seal

## VIII. References

United States Environmental Protection Agency (USEPA). 1991. *User's Guide to the Contract Laboratory Program*. Office of Emergency and Remedial Response, Washington, D.C. (EPA/540/P-91/002). January.



## Attachment 1: Sample Label and Completion Instructions

Project Name: _____ Project No: _____
Sample ID: _____
Sample Date: _____ Sample Time: _____
Sampler(s): _____
Analyses: _____
Preservatives: _____

### Completion Instructions:

The following information shall be recorded with a waterproof marker on each label:

- Project name
- Project number
- Sample identification or number
- Date and time of sample collection (24 hour clock)
- Sampler's name or initials
- Sample preservatives (if applicable)
- Analyses to be performed on the sample (specifically for the specific container and preservatives – typically for water samples only). This shall be identified by the method number (or name if the number is not known).

These labels may be obtained from the analytical laboratory or printed from a computer onto adhesive labels.





## Attachment: Custody Label and Completion Instructions

Signature: _____
Date and time _____ Time: _____

### Completion Instructions:

The custody seals shall contain the following information:

- Signature
- Date and time the sample container (bottle/jar/vial) or sample shipping container (cooler) was sealed (24 hour clock)

The custody seals may be obtained from the analytical laboratory or printed from a computer onto adhesive labels.

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**Standard Operating Procedure - 18**

Title: **Equipment Blank and Field Blank Preparation**

Document Number: **SOP 18**

**Contract No: N62473-09-D-2622**

**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Equipment Blank and Field Blank Preparation

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## I. Purpose

To prepare blanks to determine whether decontamination procedures are adequate and whether any cross-contamination is occurring during sampling due to contaminated air and dust.

## II. Scope

The general protocols for preparing the blanks are outlined. The actual equipment to be rinsed will depend on the requirements of the specific sampling procedure.

## III. Equipment and Materials

- Blank liquid (use ASTM Type II or lab-grade water)
- Distilled water
- Sample bottles as appropriate
- Gloves
- Preservatives as appropriate

## IV. Procedures and Guidelines

1. Decontaminate all sampling equipment that has come in contact with the sample according to SOP *Decontamination of Personnel and Equipment*.
2. To collect an equipment blank for analysis of volatile organic compounds (VOC) or total petroleum hydrocarbons-purgeable (TPH-p) from the surfaces of sampling equipment, pour blank water over one piece of equipment and into three 40-milliliter (ml) vials (per analysis) until there is a positive meniscus, then seal the vials. Note the sample number and associated piece of equipment in the field notebook as well as the type and lot number of the water used.

For analyses of nonvolatile compounds, one aliquot will be collected for each chemical group to be analyzed using a container type and size appropriate for each type of equipment used. For example, if a pan and trowel are used, place trowel in pan and pour blank fluid in pan such that pan and trowel surfaces that contacted the sample are contacted by the blank fluid. Pour blank fluid from pan into appropriate sample bottles.

Do not let the blank fluid come in contact with any equipment that has not been decontaminated.

3. When collecting an equipment blank from a submersible pump (Grundfos or similar), run an extra gallon of distilled water through the pump while collecting the pump outflow into appropriate containers. Make sure the flow rate is low when sampling VOCs. If a Grundfos pump with disposable tubing is used, remove the disposable tubing after sampling but before decontamination. When decontamination is complete, put a 3- to 5-foot segment of new tubing onto the pump to collect the equipment blank.
4. To collect a field blank, slowly pour ASTM Type II or lab-grade water directly into sample containers. Field blanks are prepared in the same location and field conditions as the samples to emulate the ambient conditions in which the samples were collected.

5. Document and ship samples in accordance with the procedures for other samples.
6. Collect next field sample.

## **V. Attachments**

None.

## **VI. Key Checks and Items**

- Wear a clean pair of sampling gloves for each separate equipment blank sample.
- Do not use any non-decontaminated equipment to prepare blank.
- Use ASTM-Type II or lab-grade water.



# Standard Operating Procedure - 19

Title: **Civil Surveying**

Document Number: **SOP 19**

**Contract No: N62473-09-D-2622**

**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
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12/19/14

Date

Quality Review by:

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12/19/14

Date

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# Civil Surveying

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## I. Purpose and Scope

The standard operating procedure (SOP) document describes survey procedures to be used on Navy Comprehensive Long-Term Environmental Action — Navy CLEAN projects. Modified third-order survey procedures will be used for most surveying. Where surveys that are more accurate are required, this SOP will be modified as part of the specific statement of work. Where acceptable satellite visibility is available the appropriate Global Positioning System (GPS) techniques can be used for horizontal control, measurement of horizontal coordinates, and setting vertical control. For the measurement of monitoring well elevations, GPS cannot be used and elevation values shall be measured using differential leveling methods.

## II. Records and Definitions

All field notes shall be kept in bound books. Each book shall have an index page. Each page of field notes shall be numbered and dated and shall show the initials of all crewmembers. The person taking field notes will be identified in the log. Information on weather (such as wind speed and direction or cloud cover) and on other site conditions shall also be entered in the notes. Notes shall also include instrument field identification number and environmental settings. Graphite pencils or waterproof ballpoint pens shall be used. Erasing is not acceptable; use a single-strike-through and initial it. The note keeping format shall conform to the SOP *Preparing Field Log Books*. Field notebooks shall be available onsite.

A survey report and a survey work map containing control points found and set, points surveyed, and coordinate grid lines at the required map scale shall be prepared and delivered for all survey field work performed. The survey report, which shall include copies of the field notes, shall be provided as a deliverable for project documentation. The survey report shall document the horizontal coordinate system and horizontal and vertical datum(s), the control monuments recovered, set, and used as a basis for the survey and all other features and points surveyed. The survey report shall describe the equipment and methodology used to perform the work. The survey report will also describe the results of the survey and accuracies obtained. The report shall document quality control procedures performed and their results. The report shall contain a coordinate point listing for all points surveyed. The survey report shall be provided in both paper and electronic format. The report shall be stamped, dated, and signed by the licensed land surveyor in responsible charge who shall certify that the work was completed in compliance with the specifications and that the deliverables meet or exceed (are better than) the required accuracy.

The following terms are defined to clarify discussion in this SOP:

- North American Datum of 1983 (NAD83) - The standard geodetic horizontal datum for the North American continent. The current datum is the North American Datum of 1988 (NAD83).
- National American Vertical Datum (NAVD88) - The vertical-control datum used by the National Geodetic Survey for vertical control. The current vertical datum is the North American Vertical Datum of 1988 (NAVD88).
- Horizontal Control - Horizontal location of physical monuments to be used as the horizontal basis of a survey. For Navy CLEAN projects, these shall be existing or new monuments in the immediate site area. These monuments shall be based on the NAD83 datum.

- Horizontal coordinates (X, Y) shall be reported in the State Plane Coordinate System of the State the work is being performed in and in the appropriate Zone. The units of measure (U.S. Survey feet or International feet) shall be as required by state law.
- Vertical Control - Vertical location of physical monuments to be used as the vertical basis of a survey. For Navy CLEAN projects, these shall be existing or new monuments in the immediate vicinity of the project site. These monuments shall be based on the NAVD88 vertical datum.
- ACCURACY – The appropriate GPS or Total Station and Differential Level Loop survey methods shall be used to achieve the following accuracy requirements:
  - **Horizontal Control** shall comply with third Order Class I (1:10,000) or better, as outlined in the FGDC Geospatial Positioning Accuracy Standards, Part 4: Standards for Architecture, Engineering, Construction (A/E/C), and Facility Management. For horizontal control points set utilizing RTK-Net or OPUS Static GPS methods, the horizontal positional accuracy shall be +/- 2 cm + 2 PPM or better or the baseline utilized for the point. Each point shall be checked with a significantly different satellite constellation.
  - **Vertical Control** shall be Third Order (0.05vm) or better, as outlined in the FGDC Geospatial Positioning Accuracy Standards, Part 4: Standards for Architecture, Engineering, Construction (A/E/C) and Facility Management. For vertical control points set utilizing RTK-Net or OPUS Static GPS methods, the vertical positional accuracy shall be +/- 4 cm + 2 PPM for the baseline utilized for the point. Each point shall be checked with a significantly different satellite constellation.
  - **All Other Surveys** (other than control surveys), shall comply with FGDC Geospatial Positioning Accuracy Standards, Part 4: Standards for Architecture, Engineering, Construction (A/E/C), and Facility Management with relative accuracy tolerances of +/-0.05-foot for the horizontal, +/-0.03-foot for the vertical on all other hard surfaces, and +/-0.10-foot for the vertical on soft or natural ground surfaces.

### III. Surveying

#### A. Horizontal Survey

If existing monuments are not recovered on or very near the site two indivisible pairs (three monuments semi-permanent), control monuments shall be set (e.g., 30-inch or longer, #5 re-bar or larger, or equivalent driven to refusal and appropriately capped and marked). These monuments will be set at ground level and shall achieve the accuracies stated above.

#### GPS Surveys

CORS, RTK Networks, and OPUS GPS control surveys shall comply with the processes and procedures identified in the U.S. Army Corps of Engineers, Control and Topographic Surveying Engineer Manual, EM 1110-1 -1005, Chapter 6, Planning and Conducting Control and Topographic Surveys, paragraphs 6-10 and 6-11 and Chapter 9, GPS Real Time Kinematic Topographic Survey Procedures. Each point shall be checked with a significantly different satellite constellation. During RTK collection of topographic/feature point check shots shall be taken on a project control point and documented every two hours during data collection. RTK GPS surveys cannot be used to obtain elevations (Z coordinates) for monitoring wells.

#### Total Station Surveys

For horizontal control surveys, horizontal angular measurements shall be made with a 5-second or better total station. When using a 5-second instrument the horizontal angles shall be turned four times (two each direct and inverted) with the mean of the fourth angle being within 3 seconds of the

mean of the second angle. When using a 3-second or better instrument, the angles shall be doubled (once each direct and inverted), with the mean of the second angle within 2 seconds of the first angle. The preferred minimum length of any traverse leg shall be 300 feet.

Distance measurements shall be made with a calibrated steel tape corrected for temperature and tension or a Total Station. When using a Total Station, the parts per million (ppm), curvature, and refraction corrections shall be made. Vertical angle measurements used for distance slope corrections shall be recorded to the nearest 5 seconds of arc deviation from the horizontal plane. Horizontal locations will be surveyed to within 0.05-foot of the true location.

Horizontal traverse stations shall be established and referenced for future use. All stations shall be described in the field notes with sufficient detail to facilitate their recovery later. The station shall consist of a semi-permanent mark scribed on facilities such as sidewalks, curbs, concrete slabs, or iron rod and cap.

The horizontal position of topographic and planimetric features located by Total Station surveys shall comply with the processes and procedures identified in the U.S. Army Corps of Engineers, Control and Topographic Surveying Engineer Manual, EM 1110-1 -1005, Chapter 8, Total Station Topographic Survey Procedures.

These methods shall be used in particular for determining the coordinates of surface-water and sediment sampling locations, and may be used for determining the locations of piezometers and monitoring wells. GPS surveys shall be performed by staff trained in the use of the equipment and will conform to guidance provided in this SOP and by the manufacturer.

## **B. Vertical Survey**

If existing vertical monuments are not recovered on or very near the site two pairs of semi-permanent vertical temporary benchmark (TBMs) control monuments shall be set. Where site conditions do not provide more suitable locations, the monuments shall be 30 inches or longer, #5 re-bar or larger, or equivalent driven to refusal and appropriately capped and marked. These monuments will be set at ground level and shall achieve the accuracies stated above. These monuments can be the same points as the horizontal control monuments if there are no existing features or structures available that would provide more stability for vertical control monuments (e.g., bridge headwalls, building foundation, sidewalks, or curbs where a chiseled square could be generated). Vertical control and elevation surveys shall be referenced to NAVD88. If practical, level circuits should close on a known benchmark other than the starting benchmark. The following criteria shall be met in conducting differential level loop surveys:

- Instruments shall be pegged weekly or after any time it is dropped or severely jolted.
- Foresight and backsight distances shall be reasonably balanced and shall not be greater than 250 feet in length.
- No side shot shall be used as a beginning or ending point in another level loop.
- Rod readings shall be made to 0.01-foot and estimated to 0.005-foot.
- Elevations shall be adjusted and recorded to 0.01-foot.
- Monitoring well and piezometer elevation points (except ground points) shall be obtained as turning points in a level loop.

TBMs shall be referenced and documented for future use. All TBMs shall be described in the field notes with sufficient detail to facilitate their recovery later. The TBMs shall consist of a permanent mark scribed on facilities such as sidewalks, curbs, concrete slabs, spikes set in the base of trees (not

power poles), or tops of anchor bolts for transmission line towers. (Horizontal traverse stations will not be considered as a TBM, but may be used as a level loop turning point.)

As identified for horizontal surveys RTK GPS survey methodologies can be used to obtain topographic and feature elevation data when performed according to the processes and procedures, as identified above for horizontal GPS surveys. As described above, RTK GPS surveys cannot be used to obtain elevations (Z coordinates) for monitoring wells. For the non-ground monitoring well elevation values, the RTK methods shall produce the relative accuracy identified above for "All Other Surveys."

### C. Traverse Computations and Adjustments

Traverses will be closed and adjusted in the following manner:

- Step One—Coordinate closures will be computed using unadjusted bearings and unadjusted field distances.
- Step Two—Coordinate positions will be adjusted (if the traverse closes within the specified limits) using the Least Squares adjustment method. If the traverse does not close or the GPS position does not compare within the required amounts the traverse shall be re-run or the GPS measurements shall be re-measured until they do.
- Step Three—Final adjusted coordinates will be labeled as "adjusted coordinates." Field coordinates should be specifically identified as such.
- Step Four—The direction and length of the unadjusted error of closure, the ratio of error, and the method of adjustment shall be documented along with the final adjusted coordinates.

### D. Level Circuit Computations and Adjustments

Level circuits will be closed and adjusted in the following manner:

- For a single circuit, elevations will be adjusted proportionally, provided the raw closure is within the prescribed limits for the circuit.
- In a level net where the elevation of a point is established by more than one circuit, the method of adjustment should consider the length of each circuit, the closure of each circuit, and the combined effect of all the separate circuit closures on the total net adjustments. The chosen adjustment method and both the un-adjusted and adjusted values shall be documented.

### E. Piezometer and Monitoring Well Surveys

Piezometer and monitoring well locations shall be surveyed only after the installation of the protective casing, which is set in concrete. The relative horizontal survey accuracy is  $\pm 0.05$ -foot measured at the notch or mark on the top of the casing. The vertical survey must have a relative accuracy of  $\pm 0.03$ -foot, with elevations reported to the nearest 0.01-foot. The following two elevations will be measured at piezometers and monitoring wells:

- At the notch or mark on the top of the piezometer or well riser (not on the protective casing), this will normally be on the north side unless something precludes it being located there.
- Ground surface on the north side of the well unless something precludes it being located there.

If no notch or mark exists, the point at which the elevation was measured on the inner casing shall be described so that water-level measurements may be taken from the same location.

## **F. Soil Boring or Sampling Location Surveys**

Selected soil boring or sampling locations may be located by the survey crew after the soil borings or sampling activities are complete. The selected borings will be staked in the field by the field team leader. The stake will be marked with the boring or sampling location number for reference. The horizontal survey accuracy is  $\pm 1$  foot and is measured to any point on the ground surface immediately adjacent to the stake.

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# Standard Operating Procedure - 21

Title: **Locating and Clearing Underground Utilities**

Document Number: **SOP 21**

**Contract No: N62473-09-D-2622**  
**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
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Quality Control Manager

12/19/14

Date

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# Locating and Clearing Underground Utilities

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## I. Purpose

The purpose of this standard operating procedure (SOP) is to provide general guidelines and specific procedures that must be followed on Navy Comprehensive Long-Term Environmental Action – Navy (CLEAN) projects for locating underground utilities and clearing dig locations in order to maximize our ability to avoid hitting underground utilities and to minimize liabilities to CH2M HILL Kleinfelder, A Joint Venture (KCH) and its subcontractors and health and safety risks to our project staff.

This SOP also identifies the types of utility locating services that are available from subcontractors and the various tools that are used to locate utilities, and discusses when each type of service and tool may or may not be applicable.

## II. Scope

This procedure describes the utility clearance procedures to be followed for intrusive field activities performed under the KCH CLEAN contract. Activity or Base Realignment and Closure (BRAC) installations may provide utility locating (or dig clearance) services through the public works department, Resident Officer in Charge of Construction (ROICC) or similar organization; their involvement is typically required to obtain the dig permits required before digging or drilling. KCH experience has been that the clearance services provided by the Navy do not typically meet the standards considered by the Program to be adequate, as they often simply rely on available base maps to mark utilities and do not verify utility locations using field geophysics.

While the Navy's process may provide some protection from liability for property damage, it provides neither adequate protection from health risks for KCH staff and subcontractors, nor compensation for down time should a utility be damaged as part of field activities.

The scope of services performed by utility location subcontractors can involve utility marking/mapping, the clearing of individual dig locations, or marking of utilities within a specified area.

The appropriate requested scope of services for a project will depend on the project. Clearing individual boreholes is often less expensive and allows the sub to concentrate their efforts on a limited area. However, if the scope of the investigation is not precisely defined (i.e., all borehole/ excavation locations are not pre-determined) it may be best to mark and map the vicinity of where the intrusive activities are planned. In some cases, it may be justified to mark and map the entire site area. If there is a potential for borehole/excavation locations to be added to the work (e.g., "step-out" or contingent borings) it is recommended to keep the subcontractor on call to clear additional locations as they are identified.

Clearance of individual dig locations should be done to a minimum 20-foot radius around the location.

An example SOW for utility location subcontractor procurement is provided in Attachment A.

### III. Services and Equipment

This section provides a general description of the services available to locate subsurface utilities and describes the types of equipment that these services may use to perform their work. It identifies the capabilities of each type of equipment to help the PM specify what they should require from utility location subcontractors.

#### Services

The services that are available to KCH for identifying and marking underground utilities are:

- The local public/private utility-run service, such as Underground Service Alert (USA Dig)
- Utility location subcontractor (contracted by KCH)

#### Equipment

Attachment B provides a summary of the various types of equipment used for subsurface utility location. It describes the capabilities and limitations of each in order to help the PM determine if the equipment being used by a subcontractor is appropriate and/or adequate.

It is important to make the potential subcontractors aware of the possible types of utilities (and utility materials) that are known at the site, and to have them explain in their bid what types of equipment they will use to locate utilities and clear dig locations, and what the limitations of these equipment are.

### IV. Procedures and Guidelines

This section presents specific procedures to be followed for the utility location work to be conducted by KCH and our subcontractors. In addition, a PM will have to follow the procedures required by the Activity to obtain their approvals, clearances, and dig permits where necessary. These “dig permit” requirements vary by Activity and must be added to the project-specific utility location procedures or project instructions. It is preferable that the Activity perform their clearance processes before we conduct our clearance work.

#### Activity Notification and Dig Permit Procedures

Identify Activity-specific permit and/or procedural requirements for excavation and drilling activities. Contact the Base Civil Engineer, ROICC, or Environmental Project Office (or similar) to obtain the appropriate form to begin the clearance process.

#### Utility Clearance Procedures

Do not begin subsurface construction activities (e.g., trenching, excavation, drilling, etc.) until a check for underground utilities and similar obstructions has been conducted by KCH (via utility location subcontractor) as a follow-up to the services provided by the Navy. The use of as-built drawings and utility company searches must be supplemented with a geophysical or other survey by a qualified, independent utility location subcontractor (subcontracted to KCH) to identify additional and undiscovered buried utilities.

Examples of the type of geophysical technologies include:

- **Ground Penetrating Radar (GPR)**, which can detect pipes (including gas pipes), tanks, conduits, and cables, both metallic and non-metallic, at depths up to 30 feet, depending

on equipment. Sensitivity for both minimum object size and maximum depth detectable depends on equipment selected, soil conditions, etc.

- **Radio Frequency (RF)** involves inducing an RF signal in the pipe or cable and using a receiver to trace it. Some electric and telephone lines emit RF naturally and can be detected without an induced signal. This method requires knowing where the conductive utility can be accessed to induce RF field if necessary.
- **Dual RF**, a modified version of RF detection using multiple frequencies to enhance sensitivity but with similar limitations to RF
- **Ferromagnetic Detectors** are metal detectors that will detect ferrous and non-ferrous utilities. Sensitivity is limited, e.g., a 100-mm iron disk to a depth of about one meter or a 25-mm steel paper clip to a depth of about 20 cm.
- **Electronic markers** are emerging technologies that impart a unique electronic signature to materials such as polyethylene pipe to facilitate location and tracing after installation. Promising for future installations but not of help for most existing utilities already in place.

The following procedures shall be used to identify and mark underground utilities during subsurface construction activities on the project:

- Contact utility companies or the state/regional utility protection service (such as USA Dig) at least five (5) working days prior to intrusive activities to advise of the proposed work, and ask them to establish the location of the utility underground installations prior to the start of actual excavation: this is a law. These services will only mark the location of public-utility-owned lines and not Navy-owned utilities. In many cases, there will not be any public-utility-owned lines on the Activity. There may also be Base-access issues to overcome.
- Procure and schedule the utility location subcontractor.
- The utility location subcontractor shall determine the most appropriate geophysical technique or combinations of techniques to identify the buried utilities on the project site, based on the survey contractor's experience and expertise, types of utilities anticipated to be present and specific site conditions. *The types of utilities must be provided to the bidding subcontractors in the SOW and procedures to be used must be specified by the bidder in their bid. It is extremely helpful to provide the sub with utility maps, with the caveat that all utilities are not necessarily depicted.*
- The utility location subcontractor shall employ the same geophysical techniques used to identify the buried utilities, to survey the proposed path of subsurface investigation/construction work to confirm no buried utilities are present.
- Obtain utility clearances for subsurface work on both public and private property.
- Clearances provided by the KCH-subcontracted service are to be in writing, signed by the party conducting the clearance. The KCH utility location subcontractor shall be required to fill out the form provided in Attachment C (this can be modified for a particular project as necessary) indicating that each dig/drill location has been addressed. The completed form shall be submitted back to KCH field staff or project manager within 24 hours of completing the utility locating activities. *This documentation requirement (with a copy of the form) needs to be provided in the subcontractor SOW.* USA Dig Alert provides a list of utilities that it will notify when it

issues a work request ticket. A copy of the USA Dig Alert work request ticket and any email communication with USA Dig Alert will be printed and kept on file.

- Utility marking shall be done using the color coding presented in Attachment D. The type of material used for marking must be approved by the Activity prior to marking. Some base commanders have particular issues with persistent spray paint on their sidewalks and streets. *Any particular marking requirements need to be provided in the subcontractor SOW.*
- Protect and preserve the markings of approximate locations of facilities until the markings are no longer required for safe and proper excavations. If the markings of utility locations are destroyed or removed before excavation commences or is completed, the Project Manager must notify the utility company or utility protection service to inform them that the markings have been destroyed.
- Perform a field check prior to drilling/digging (preferably while the utility location sub is still at the site) to see if field utility markings coincide with locations on utility maps. Look for fire hydrants, valves, manholes, light poles, lighted signs, etc to see if they coincide with utilities identified by the subcontractor.
- Underground utility locations must be physically verified (or dig locations must be physically cleared) by hand digging to a depth of at least five feet using a hand auger, wood or fiberglass-handled tools, air knifing, or by some other acceptable means approved by KCH, when the dig location (e.g., mechanical drilling, excavating) is expected to be within 5 feet of a marked underground system. Hand clearance shall be conducted to the depth of the deepest known or suspected utilities and no less than five feet below ground surface. Hand clearance to deeper than five feet shall be conducted if utility clearance or utility survey information indicated the presence of utilities at greater depth.
- Conduct a site briefing for employees at the start of the intrusive work regarding the hazards associated with working near the utilities and the means by which the operation will maintain a safe working environment. Detail the method used to isolate the utility and the hazards presented by breaching the isolation.
- Monitor for signs of utilities during advancement of intrusive work (e.g., sudden change in advancement of auger or split spoon during drilling or change in color, texture, or density during excavation that could indicate the ground has been previously disturbed).

## V. Attachments

A – Example SOW for Utility Location Subcontractor Procurement

B – Equipment Used for Identifying Underground Utilities

C – Buried Utility Location Tracking Form

D – Utility Marking Color Codes

# Attachment A – Example SOW for Subcontracting Underground Utilities Locating Services

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CTO-XXX  
Scope of Work  
Subsurface Utility Locating  
Site XX  
Navy Activity

## City, State

A licensed and insured utility clearance subcontractor (Subcontractor) will be subcontracted to identify and mark out subsurface utilities for an environmental investigation/remediation project at Site XX of <<insert name of base, city, and state>>. The Subcontractor will need to be available beginning at <<insert time>> on <<insert date>>. It is estimated that the work can be completed within XX days.

### Proposed Scope of Work

The Subcontractor will identify and mark all subsurface utilities (CHOOSE 1) that lie within a radius of 20 feet of each of XX sampling locations at Site XX shown on the attached Figure 1; (OR) that lie within the bounds of Site XX as delineated on the attached Figure 1. (If multiple sites are to be cleared, provide maps of each site with sample locations or clearance boundaries clearly delineated and a scale provided.)

Utilities will be identified using all reasonably available as-built drawings, electronic locating devices, and any other means necessary to maintain the safety of drilling and sampling personnel and the protection of the base infrastructure. The location of utilities identified from as-built drawings or other maps must be verified in the field prior to marking.

Base utility drawings for the Site(s) (CHOOSE 1) can be found at <<insert specific department and address or phone number on the base>> and should be reviewed by the subcontractor and referenced as part of the utility locating, (OR) will be provided to the subcontractor by KCH upon the award of the subcontract, (OR) are not available. Utility drawings shall not be considered definitive and must be field verified.

Field verification will include detection using nonintrusive subsurface detection equipment (magnetometers, GPR, etc) as well as opening manhole covers to verify pipe directions. As part of the bid, the Subcontractor shall provide a list of the various subsurface investigation tools they propose to have available and use at the site and what the limitations are of each tool.

A KCH representative shall be present to coordinate utility clearance activities and identify points and features to be cleared.

### Field Marking and Documentation

All utilities located within (CHOOSE 1) a 20-ft radius of the XX proposed soil boring locations (OR) within the boundary of the site(s) as identified on the attached figure(s) will be marked using paint (some Bases such as the WNY may have restrictions on the use of permanent paint) and/or pin flags color coded to indicate electricity, gas, water, steam, telephone, TV cable, fiber optic, sewer, etc. The color coding shall match the industry standard as described on the attached form. In addition, the Buried Utility Location Tracking Form (Attachment C) will be completed by the

Subcontractor based upon what is identified in the field during the utility locating and submitted back to KCH (field staff or project manager) within 24 hours of completing the utility locating activities.

(OPTIONAL) The subcontractor shall also provide a map (or hand sketch) of the identified utilities to the Engineer within XX days of field demobilization. The map shall include coordinates or ties from fixed surface features to each identified subsurface utility.

### **Bid Sheet/Payment Units**

The Subcontractor will bid on a time and materials basis for time spent on site and researching utility maps. Mobilization (including daily travel to the site) should be bid as a lump sum, as well as the preparation of the AHA and any required mapping. The per diem line item should be used if the field crew will require overnight accommodations at the project site.

### **Health and Safety Requirements**

The **Subcontractor** is to provide and assume responsibility for an adequate corporate Health and Safety Plan for onsite personnel. Standard personal safety equipment including: hard hat, safety glasses, steel-toed boots, gloves are recommended for all project activities. Specific health and safety requirements will be established by the Subcontractor for each project. The health and safety requirements will be subject to the review of KCH.

The **Subcontractor** shall also prepare and provide to the Engineer, at least 48 hours prior to mobilization, an acceptable Activity Hazard Analysis (AHA) using the attached AHA form or similar.

It is also required that all **Subcontractor** personnel who will be on site attend the daily health and safety tailgate meeting at the start of each day in the field.

Subcontractor personnel showing indications of being under the influence of alcohol or illegal drugs will be sent off the job site and their employers will be notified. Subcontractor personnel under the influence of prescription or over-the-counter medication that may impair their ability to operate equipment will not be permitted to do so. It is expected that the **Subcontractor** will assign them other work and provide a capable replacement (if necessary) to operate the equipment to continue work.

### **Security**

The work will typically be performed on US Navy property. KCH will identify the Subcontractor personnel who will perform the work to the appropriate Navy facility point-of-contact, and will identify the Navy point-of-contact to the Subcontractor crew. The Subcontractor bears final responsibility for coordinating access of his personnel onto Navy property to perform required work. This responsibility includes arranging logistics and providing to KCH, in advance or at time of entry as specified, any required identification information for the Subcontractor personnel. Specifically, the following information should be submitted with the bid package for all personnel that will perform the work in question (this information is required to obtain a base pass):

- Name
- Birth Place
- Birth Date
- Social Security Number
- Drivers License State and Number
- Citizenship

Please be advised that no weapons, alcohol, or drugs will be permitted on the Navy facility at any time. If any such items are found, they will be confiscated, and the Subcontractor will be dismissed.

### **Quality Assurance**

The Subcontractor will be licensed and insured to operate in the State of <<state>> and will comply with all applicable federal, state, county and local laws and regulations. The subcontractor will maintain, calibrate, and operate all electronic locating instruments in accordance with the manufacturer's recommendations. Additionally, the Subcontractor shall make all reasonable efforts to review as-built engineering drawings maintained by Base personnel, and shall notify the KCH Project Manager in writing (email is acceptable) whenever such documentation was not available or could not be reviewed.

### **Subcontractor Standby Time**

At certain periods during the utility locating activities, the Subcontractor's personnel may be asked to stop work and standby when work may normally occur. During such times, the Subcontractor will cease activities until directed by the KCH representative to resume operations. Subcontractor standby time also will include potential delays caused by the KCH representative not arriving at the site by the agreed-upon meeting time for start of the work day. Standby will be paid to the Subcontractor at the hourly rate specified in the Subcontractor's Bid Form attached to these specifications.

Cumulative Subcontractor standby will be accrued in increments no shorter than 15 minutes (i.e., an individual standby episode of less than 15 minutes is not chargeable).

During periods for which standby time is paid, the surveying equipment will not be demobilized and the team will remain at the site. At the conclusion of each day, the daily logs for the Subcontractor and KCH representative will indicate the amount of standby time incurred by the Subcontractor, if any. Payment will be made only for standby time recorded on KCH's daily logs.

### **Down Time**

Should equipment furnished by the Subcontractor malfunction, preventing the effective and efficient prosecution of the work, or inclement weather conditions prevent safe and effective work from occurring, down time will be indicated in the Subcontractor's and KCH representative's daily logs. No payment will be made for down time.

### **Schedule**

It is anticipated that the subsurface utility locating activities will occur on <<insert date>>. It is estimated that the above scope will be completed within XXX days.

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# Attachment B – Equipment Used for Identifying Underground Utilities

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This attachment provides a summary of the various types of equipment used for subsurface utility location. It describes the capabilities and limitations of each in order to help determine if the equipment being proposed by a subcontractor or Navy is adequate.

## Electromagnetic Induction (EMI) Methods

EMI instruments, in general, induce an electromagnetic field into the ground (the primary field) and then record the response (the secondary field), if any. Lateral changes in subsurface conductivity, such as those caused by the presence of buried metal or by significant soil variations, cause changes in the secondary field recorded by the instrument and thus enable detection and mapping of the subsurface features. It should be noted that EMI only works for electrically conductive materials--plastic or PVC pipes are generally not detected with EMI. Water and gas lines are commonly plastic, although most new lines include a copper "locator" strip on the top of the PVC to allow for detection with EMI.

EMI technology encompasses a wide range of instruments, each with inherent strengths and weaknesses for particular applications. One major division of EMI is between "time-domain" and "frequency-domain" instruments that differ in the aspect of the secondary field they detect. Another difference in EMI instruments is the operating frequency they use to transmit the primary field. Audio- and radio-frequencies are often used for utility detection, although other frequencies are also used. Consideration of the type of utility expected, surface features that could interfere with detection, and the "congestion" of utilities in an area, should be made when choosing a particular EMI instrument for a particular site.

One common EMI tool used for utility location is a handheld unit that can be used to quickly scan an area for utilities and allows for marking locations in "real time." This method is most commonly used by "dig-safe" contractors marking out known utilities prior to excavation. It should be noted that this method works best when a signal (the primary field) can be placed directly onto the line (i.e., by clamping or otherwise connecting to the end of the line visible at the surface, or for larger utilities such as sewers, by running a transmitter through the utility). These types of tools also have a limited capability to scan an area for unknown utilities. Usually this requires having enough area to separate a hand held transmitter at least a hundred feet from the receiver. Whether hunting for unknown, or confirming known, utilities, this method will only detect continuous lengths of metallic conductors.

In addition to the handheld EMI units, larger, more powerful EMI tools are available that provide more comprehensive detection and mapping of subsurface features. Generally, data with these methods are collected on a regular grid in the investigation area, and are then analyzed to locate linear anomalies that can be interpreted as utilities. These methods will usually detect *all* subsurface metal (above a minimum size), including pieces of abandoned utilities. In addition, in some situations, backfill can be detected against native soils giving information on trenching and possible utility location. Drawbacks to these methods are that the secondary signals from utilities are often swamped (i.e., undetectable) close to buildings and other cultural features, and that the subsurface at heavily built-up sites may be too complicated to confidently interpret completely.

Hand-held metal detectors (treasure-finders) are usually based on EMI technology. They can be used to locate shallow buried metal associated with utilities (e.g., junctions, manholes, metallic

locators). Advantages of these tools are the ease of use and real-time marking of anomalies. Drawbacks include limited depths of investigations and no data storage capacity.

### **Ground Penetrating Radar (GPR)**

GPR systems transmit radio and microwave frequency (e.g., 80 megaHertz to 1,000 megaHertz) waves into the ground and then record reflections of those waves coming back to the surface. Reflections of the radar waves typically occur at lithologic changes, subsurface discontinuities, and subsurface structures. Plastic and PVC pipes can sometimes be detected in GPR data, especially if they are shallow, large, and full of a contrasting material such as air in a wet soil, or water in a dry soil. GPR data are usually collected in regular patterns over an area and then analyzed for linear anomalies that can be interpreted as utilities. GPR is usually very accurate in x-y location of utilities, and can be calibrated at a site to give very accurate depth information as well. A significant drawback to GPR is that depth of investigation is highly dependent on background soil conductivity, and it will not work on all sites. It is not uncommon to get only 1-2 feet of penetration with the signal in damp, clayey environments. Another drawback to GPR is that sites containing significant fill material (e.g., concrete rubble, scrap metal, garbage) will result in complicated anomalies that are difficult or impossible to interpret.

### **Magnetic Field Methods**

Magnetic field methods rely on detecting changes to the earth's magnetic field caused by ferrous metal objects. This method is usually more sensitive to magnetic metal (i.e., deeper detection) than EMI methods. A drawback to this method is it is more susceptible to being swamped by surface features such as fences and cars. In addition, procedures must usually be implemented that account for natural variations in the earth's background field as it changes throughout the day. One common use of the method is to measure and analyze the gradient of the magnetic field, which eliminates most of the drawbacks to the method. It should be noted this method only detects ferrous metal, primarily iron and steel for utility location applications. Some utility detector combine magnetic and EMI methods into a single hand-held unit.

### **Optical Methods**

Down-hole cameras may be useful in visually reviewing a pipe for empty conduits and/or vaults.

# Attachment C – Buried Utility Location Tracking Form

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See next page.

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# Attachment D – Utility Marking Color Codes

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The following is the standard color code used by industry to mark various types of utilities and other features at a construction site.

White – Proposed excavations and borings

Pink – Temporary survey markings

Red – Electrical power lines, cables, conduits and lighting cables

Yellow – Gas, oil, steam, petroleum, or gaseous materials

Orange – Communication, alarm or signal lines, cables, or conduits

Blue – Potable water

Purple – Reclaimed water, irrigation, and slurry lines

Green – Sewer and storm drain lines

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**Standard Operating Procedure - 23**

Title: **Trenching for Landfill Delineation**

Document Number: **SOP 23**

**Contract No: N62473-09-D-2622**

**NAVFAC Southwest CLEAN IV**

Technical Review by:

Douglas Gilkey  
Technical Reviewer

12/19/14

Date

Quality Review by:

Theresa Rojas  
Program Quality Assurance/  
Quality Control Manager

12/19/14

Date

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# Trenching for Landfill Delineation

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## I. Purpose

To provide reference material and general guidance on test pitting using a backhoe during landfill boundary confirmation activities. This standard operating procedure (SOP) will guide staff in investigation procedures for consistent documentation of subsurface conditions. Additional applicable procedures include utility clearance, soil logging, and health and safety procedures for test pits. Procedures associated with soil sampling, munitions or concern, unexploded ordnance, or radiological screening may also be applicable as discussed in the work plan.

## II. Equipment and Materials

- Site map with test pit locations and associated global positioning system (GPS) coordinates
- Work Plan for Field Investigation including Health and Safety Plan
- Backhoe (to be supplied by others)
- Shovels, picks, and scoops
- Gallon zip-lock plastic bags (for collecting waste samples, if necessary)
- Tape measure and measuring wheel
- Hand-held GPS (preferably with test pit coordinates loaded)
- 48-inch survey lath stakes and rolls of plastic flagging
- Permanent marking pen (Sharpie) for lath stakes
- Digital camera with flash for illuminating shaded trench walls
- Flashlight or mirror (also for illuminating shaded trench walls)
- Dry-erase board (or other erasable material)
- Plastic sheeting and stakes and/or sand bags for placement and cover of excavated soil
- Barricades to secure the area around the excavation
- Onsite decontamination pad
- Photoionization detector organic vapor monitor (OVM)
- Gas monitor for oxygen, lower explosive limit, methane, and carbon dioxide
- Level D personnel protection equipment (or higher protective levels as required) and onsite site safety officer

## III. Procedures and Guidelines

### General Considerations

All work is to be performed in accordance with the Accident Prevention Plan/Site –specific Safety and Health Plan (APP/SSHP) and meet all legal requirements for trenching and excavating (including but not limited to safety, training, utility clearance, and shoring).

After completion of buried utility clearance activities (KCH SOP 21), landfill delineation test pits will be excavated along the landfill boundary as indicated by the results of soil borings and/or geophysical surveys.

Test pit locations and depths shall be excavated as described in the work plan. Modifications to the locations and depths of test pits and trenches will likely be necessary based on field observations. Adjustments to planned locations and depths shall be discussed and agreed to with the Task Order Manager.

Conduct test pitting following this general procedure:

1. Set up at estimated edge of waste (as described “Trenching Activities” items 2 through 5 below).
2. Excavate until waste is encountered or a depth of 10 feet is reached, as feasible based on trench safety considerations.
3. If waste is encountered, extend trench radially away from the landfill following the cover soil/waste interface. If the edge of waste is not encountered after extending the trench outwards a distance of 15 feet, step out radially an additional 20 feet and excavate a new trench to a similar depth. If waste is encountered at the new location, step out another 20 feet. If not, the edge of the waste material has been passed, therefore step 10 feet back towards the landfill, and excavate to a similar depth and advance trench towards the end of the initial trench or the second trench depending on if waste is encountered.
4. If waste is not encountered in the initial trench (#2 above), step radially 20 feet towards the landfill and excavate to waste or until a depth of 10 feet is reached. If waste is found, extend the trench laterally towards the initial trench, following cover soil/waste interface. If waste is not encountered, repeat the step process.
5. When the edge of waste is found, document locations of trenches and the edge of the waste. If using a GPS, obtain a location of both ends of each trench excavated as well as the edge of the waste. If marking locations for a later survey, set flagged lath stakes at both ends of each trench. Label lath stakes in pairs (A and A' for trench A) to avoid confusion when trenches are closely spaced in a radial line. Also stake the edge of the waste and label the lath stake “EW” for edge of waste.

At a minimum, document the following information for each trench (in writing and with photos as appropriate):

- Thicknesses of soil cover over waste, and soil classification.
- Character of the waste (organic or inorganic, decomposed or not, dry or moist, black or some other color, describe major components and approximate percentage of waste [such as glass, metal, green waste, paper, plastic]).
- Note if waste is present as a mass or is mixed with a significant amount of soil. If the bottom of the waste is observed, check if the soil beneath the waste appears to be impacted by leachate from the waste (staining, moisture).
- If water is encountered note the depth from surrounding natural grade and note whether water appears to be perched or changes in water level during test pit excavation.

The staff will photograph and document any significant features exposed by the test pit. Photographs will be labeled with (at a minimum) the test pit number, the date and time of photograph, a description of the feature, the direction the camera was facing, and size scale. Other observations (including soil descriptions) will be recorded in the logbook.

Unless otherwise required by project specific work plans or applicable soil management plans; once test pitting is complete, the pits will be backfilled with the stockpiled soil/waste in the reverse order of removal (e.g., last out/first in) and regraded to blend with the original ground surface. The finished surface of the test pit should not create a new condition where precipitation can pond within the test pit area.

## **Trenching Activities**

1. Establish the work zone according to the site safety plan.
2. Conduct all required activities for the location and identification of buried utilities (KCH SOP 21).
3. Place plastic sheeting at the side of the planned excavation area to provide a temporary stockpile area. The temporary stockpile should be located a safe distance away from the planned excavation to avoid the risk of excavated material falling into the excavation and the risk that the stockpiled material will cause trench or test pit wall to collapse with the added load (minimum required distance 4 feet); however, site conditions and health and safety requirements may require placement of material at a greater distance from the test pit.
4. Calibrate all air quality monitoring instruments and begin air quality monitoring requirements (if any) as specified in the project specific work plan and site-specific health and safety plan.
5. Using a cleaned backhoe bucket (decontaminated following SOP 12 Decontamination of Drilling Rigs and Equipment), begin excavating towards the area defined as the landfill boundary during the geophysical survey. Place excavated soil onto the plastic sheeting to be used for temporary stockpiling.
6. As the trenching is advanced, periodically halt excavation activities to allow for photographic and written observations to be entered on the trench log. Advance trenching progressively until buried material is encountered. Remove only enough soil to identify the landfill boundary.
7. Monitor the air quality in the breathing zone continuously, with documented readings taken at no less than 5-minute intervals during trenching activities. Evacuate the trench immediately if gas-screening levels exceed safety threshold values as identified in the SSHP.
8. Photograph and document the trenching activities as required by the Work Plan data quality objectives. Backfill and regrade the test pit, dispose of used plastic sheeting in accordance with the investigation derived waste management plan, decontaminate the backhoe, and set up at the next trenching location.

## **IV. Attachments**

Trench and Test Pit Log

## **V. Key Checks and Items**

No personnel shall enter the trench or test pit unless specifically identified in the work plan. Entering a trench or test pit necessitates additional health and safety requirements.

Ensure that the excavator is fully stopped and that the backhoe bucket is resting on the ground during observation, photographing, and documentation of trench condition by staff at the edge of the excavation.

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# TEST PIT FIELD LOG

Project Name:

Project Number:

Sheet: \_\_\_\_ of \_\_\_\_

Test Pit No./Box No(s):	Location:	Excavation Equipment:
Total Depth:	Coordinates (X/Y, Lat/Long):	Excavation Company:
Date Begin/End:	Coordinate System/Datum:	Logged By/Checked By:
Surface Conditions:	TP Direction/Orientation:	Depth to Groundwater Initial/Time: Final/Time:

GRAPHIC LOG OF TEST PIT WALL  
(Depicts \_\_\_\_\_ Facing Wall)

Depth

Elevation

Scale: 1 in = ..... feet

Unit	Depth	Material Description

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**Attachment 4**  
**KCH Soil Boring Log**

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<i>Date Started</i> _____	<i>Location</i> _____
<i>Logged By</i> _____	<i>Date Completed</i> _____
<i>Total Depth</i> _____	<i>Drilling Method</i> _____
<i>Latitude</i> _____	<i>Hammer Wt</i> _____
<i>Longitude</i> _____	<i>Surface Conditions</i> _____
	<i>Surface Elevation</i> _____

Depth (feet)	Sample Number	Sample Type	Blows/Foot	Recovery (%)	OVA (ppm) PID/FID	USCS	Description	Remarks	Well Construction
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	<b>LOG OF BORING NO.</b>	<i>PLATE</i>
<i>PROJECT NO.</i>		

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**Attachment 5**  
**KCH Trench Log**

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# TEST PIT FIELD LOG

Project Name:

Project Number:

Sheet: \_\_\_\_ of \_\_\_\_

Test Pit No./Box No(s):	Location:	Excavation Equipment:
Total Depth:	Coordinates (X/Y, Lat/Long):	Excavation Company:
Date Begin/End:	Coordinate System/Datum:	Logged By/Checked By:
Surface Conditions:	TP Direction/Orientation:	Depth to Groundwater Initial/Time: Final/Time:

GRAPHIC LOG OF TEST PIT WALL  
(Depicts \_\_\_\_\_ Facing Wall)

Depth

Elevation

Scale: 1 in = \_\_\_\_\_ feet

Unit	Depth	Material Description

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**Attachment 6**  
**Groundwater Sampling Data Sheet**

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**Attachment 7**  
**Laboratory SOPs**

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SOP REVIEW FORM

EMAX-6020  
SOP No.

Rev. 8  
Revision Number

TRACE METAL BY ICP-MS  
Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

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This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

Mary Jane Mendoza *[Signature]*

Date:

7/8/14

## STANDARD OPERATING PROCEDURES

**TRACE METALS BY ICP-MS**

SOP No.: EMAX-6020 Revision No. 8 Effective Date: 27-Jun-13

Prepared By: Mary Jane Mendoza *[Signature]* Date: 06-27-13

Approved By: Kenette Pimentel *[Signature]* Date: 06-27-13  
QA Manager

Approved By: Caspar Pang *[Signature]* Date: 06-27-13  
Laboratory Director

**Control Number: 6020-08**

**1.0 SCOPE AND APPLICATION**

- 1.1. This procedure is applicable for the determination of sub- $\mu\text{g/L}$  concentrations of a large number of elements in wastewater, groundwater, aqueous, extract, soil, sludge, and sediment samples using the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) method. All matrices require proper sample preparation prior to analysis.
- 1.2. The elements and their corresponding isotopes are listed in Table 1.
- 1.3. This SOP is an adaptation of the SW846 Method 6020A.

**2.0 SUMMARY OF METHOD**

- 2.1. Metal analytes in water are acid digested from a pre-measured sample. Nitric acid and hydrochloric acid are added to the sample and heated without boiling until the volume is substantially reduced. The digestate is diluted back to its original sample volume using reagent water.
- 2.2. Metal analytes in soil are acid digested from a pre-measured sample. Nitric acid is added to the sample and heated to initialize digestion. It is further oxidized with 30% hydrogen peroxide and the acid used for final reflux is hydrochloric acid.
- 2.3. Digestates are introduced by pneumatic nebulization resulting aerosol into a high temperature argon plasma, where they are decomposed, atomized and ionized. The ions produced are extracted from the plasma via the sample and skimmer orifices in the interface region of the mass spectrometer. The extracted ions are guided by an off-axis Lens System to reduce background noise, passes through an Octopole Reaction System (ORS) where some ions require a simple reaction with  $\text{H}_2$  or He to eliminate matrix interference prior to entering the Quadrupole Mass Filter (QMF). The QMF separates ions based on their mass-to-charge ratios and ions are counted by electron multiplier detector.
- 2.4. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a calibration curve.
- 2.5. **Interference**
  - 2.5.1. Isobaric Elemental Interference. Are caused by isotopes of different elements which form singly or doubly charge ions of the same nominal mass-to-charge ratio. The signal of an isotope of an interfering element is determined and subtracted from the analyte isotope signal.
  - 2.5.2. Isobaric Polyatomic Ion Interference. Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest. To correct for isobaric polyatomic ion interferences, optimize the cell gas pressure on each analyte so that when the ORS employs simple reaction gases ( $\text{H}_2$  and He) side reactions create new and unpredictable interferences. The ORS is equipped with notch filters and by using scanning voltages the created interferences are prevented from reaching the analyzer.

## STANDARD OPERATING PROCEDURES

**TRACE METALS BY ICP-MS**SOP No.: EMAX-6020 Revision No. 8 Effective Date: 27-Jun-13

- 2.5.3. Physical Interference. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. An internal standard can be used to correct physical interference if carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of the internal standard is less than 70% of the intensity of the first standard used during calibration, the sample must be diluted and re-analyzed.
- 2.5.4. Memory Interference. Contamination by carry-over can occur whenever high concentrations are analyzed in sequence with a low concentration sample. To reduce potential carry-over the rinse period between samples must be long enough to eliminate significant memory effect.

**3.0 DETECTION LIMITS****3.1. Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Established limits are shown in Table 5.

**4.0 DYNAMIC RANGE**

- 4.1. Linear Dynamic Range (LDR) is the concentration over which the instrument response remains linear.
- 4.2. Establish LDR of each analyte by determining the signal response from a minimum of three preferably five different concentration across the range. The upper limit should be within 10% ( $\pm 10\%$ ) of the true value.
- 4.3. Verify the established LDR every six months or when there is a significant change in the instrument signal, whichever comes first.

**5.0 SAMPLE HOLDING TIME AND PRESERVATION****5.1. Holding Time**

- 5.1.1. Analyze all samples within 180 days from collection date.

**5.2. Preservation**

- 5.2.1. Expected sample condition when received in the lab:
- water samples in HDPE, preserved to pH < 2 with HNO<sub>3</sub>
  - soil samples in glass jar or brass tubes
- 5.2.2. When water sample preservation is requested to be done in the lab, preserve the sample to pH < 2 with HNO<sub>3</sub> and observe at least 24 hours from the time preservative is added before sample digestion.
- 5.2.3. Store water samples in the same condition as received unless specified in the project requirement.
- 5.2.4. Store soil samples at  $\leq 6^{\circ}\text{C}$  without freezing.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review

## STANDARD OPERATING PROCEDURES

**TRACE METALS BY ICP-MS**SOP No.: EMAX-6020 Revision No. 8 Effective Date: 27-Jun-13

- |       |           |                                   |
|-------|-----------|-----------------------------------|
| 6.2.  | EMAX-QA04 | Detection Limit (DL)              |
| 6.3.  | EMAX-QA05 | Training                          |
| 6.4.  | EMAX-QA08 | Corrective Action                 |
| 6.5.  | EMAX-QC01 | Quality Control for Chemicals     |
| 6.6.  | EMAX-QC02 | Analytical Standard Preparation   |
| 6.7.  | EMAX-QC04 | Balance Calibration               |
| 6.8.  | EMAX-QC05 | Calibration of Thermometers       |
| 6.9.  | EMAX-QC06 | Calibration of Micropipettes      |
| 6.10. | EMAX-QC07 | Glassware Cleaning                |
| 6.11. | EMAX-SM03 | Waste Disposal                    |
| 6.12. | EMAX-SM04 | Analytical and QC Sample Labeling |

**7.0 SAFETY**

- 7.1. Read all SDS of chemicals listed in this SOP.
- 7.2. Observe the following precautions during operation or maintenance of the instrument:
- Close the instruments hoods and panels prior to operation.
  - Check the exhaust system for a positive extraction at the exhaust duct.
  - Handle acids properly.
  - Check the drain vessels frequently.
  - Make sure that the argon tank is chained.
  - Wait for the instrument interface region to cool down prior to instrument maintenance.
  - Observe all cautions and warnings stipulated in the Agilent 7500 ICPMS CE/CX Manuals.
- 7.3. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Observe all chemical hygiene procedures as mentioned in the Chemical Hygiene Plan.
- 7.4. DO NOT DISPOSE ACIDIC WASTE IN THE TRASH CAN OR IN THE SINK.
- 7.5. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS AND REAGENTS****8.1. Instruments and Supplies**

- 8.1.1. ICP-MS: Agilent 7500CE Octopole Reaction System : Agilent 7500CX Octopole Reaction System
- 8.1.2. Autosampler: CETAC ASX-520
- 8.1.3. Computer: IBM Compatible

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- 8.1.4. RF Generator: Agilent RF Generators
- 8.1.5. Data Acquisition: Agilent Chemstation version B.03.04 or as updated : Agilent Chemstation version B.04.00 or as updated
- 8.1.6. Autosampler rack(s): 17 x 100 mm, 60 positions
- 8.1.7. Culture tubes: 17 x 100 mm, polypropylene
- 8.1.8. Volumetric Flask: 10 ml, 50 ml, 500 ml, 1000 ml
- 8.1.9. Micropipettes: 1 ml, 0.100 ml, 5 ml
- 8.1.10. Pipet Tips: 100 - 1000 µl
- 8.1.11. Polyethylene bottles: 250, 500, 1000 ml
- 8.1.12. Liquid argon
- 8.1.13. Hydrogen, Compressed: Ultra-high purity
- 8.1.14. Helium, compressed: Ultra-high purity
- 8.1.15. Balance: Sartorius LC 620 S or equivalent
- 8.1.16. Spatula: Stainless steel or equivalent
- 8.1.17. Digestion vessel: 50 ml, 100 ml snap seal
- 8.1.18. Digestion block: Aluminum blocks or equivalent
- 8.1.19. Thermometer: Range 0 - 110°C
- 8.1.20. Filter: Whatman #41 or equivalent
- 8.1.21. Digestate Container: 50 ml polyethylene vessel, 100 ml Corning snap seal or equivalent
- 8.1.22. Disposable watch glass
- 8.1.23. ASX press Rapid Sample Introduction System

**8.2. Chemicals and Reagents**

- 8.2.1. DI water, ASTM Type II or equivalent
- 8.2.2. Nitric Acid, Trace high purity grade, concentrated
- 8.2.3. Hydrochloric acid, Trace high purity grade, concentrated
- 8.2.4. Hydrogen Peroxide, ACS grade (e.g., VW3690-5 from VWR) or equivalent

**9.0 STANDARDS****9.1. Tune Check Standard**

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
Tuning Solution	Agilent	Li, Y, Ce, Tl, Co	0.01	2% HNO <sub>3</sub>
Tuning Check	High Purity	Co, In, Li, Tl	10	2% HNO <sub>3</sub>

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Standard	Standard			
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9.1.1. Prepare tuning check standard at concentration level suggested below.

STANDARD	Aliquot, (ml)	Final volume (ml)	Final Concentration (mg/L)
Intermediate tuning check standard	0.5	50	0.100

9.2. **Internal Standard (IS)**

9.2.1. Purchase stock internal standard as certified standard at concentration listed below or equivalent.

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
IS	Agilent	Li6, Sc, Ge, Rh, In, Tb, Bi, Lu	100	10% HNO <sub>3</sub>

9.3. **Calibration Standards**9.3.1. Calibration Stock Standard

9.3.1.1. Purchase custom-made certified individual and mixed stock standards as listed in the table below or equivalent.

STANDARD	SOURCE	ELEMENTS	CONC. (µg/ml)	MATRIX
ICAL 1	High Purity	As, B, Se, Sr, Tl, Ti, V, Zn, Sb, Mo, Sn	10	2% HNO <sub>3</sub> and Trace HF Acid
ICAL 2	High Purity	Ba, Be, Cd, Cr, Co, Cu, Pb, Li, Mn, Ni, Ag, U	10	2% HNO <sub>3</sub>
ICAL 3	High Purity	Al, Fe, K, Ca, Mg, Na	1000	4% HNO <sub>3</sub>
Phosphorus	AccuStandard	P	100	Water
Tungsten	AccuStandard	W	100	Water and trace NH <sub>4</sub> OH
Zirconium	AccuStandard	Zr	100	2-5% HNO <sub>3</sub>
Zinc	High Purity	Zn	1000	2% HNO <sub>3</sub>

9.3.2. Calibration Working Standard

9.3.2.1. Prepare working standards as suggested in the table below or equivalent.

Working Standard	Stock Standard Name	Conc. (µg/ml)	Source	Preparation		
				Aliquot (ml)	Final vol. (ml)	Final conc. (µg/ml)
Trace mix	ICAL 1	10	High Purity	1.0	10	1.0
	ICAL 2	10	High Purity	1.0		1.0
Cation Mix	Al	10,000	AccuStandard	2.5	500	50
	Ca	10,000	AccuStandard	2.5		50
	Fe	10,000	AccuStandard	2.5		50

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	Mg	10,000	AccuStandard	2.5		50
	K	10,000	AccuStandard	2.5		50
	Na	10,000	AccuStandard	1.25		25
Zn – 10 ppm	Zn	1000	High Purity	0.5	50	10
Zr – 1 ppm	Zr	100	AccuStandard	0.5	50	1.0
W – 1 ppm	W	100	AccuStandard	0.5	50	1.0
Mix 7A	ICAL 1	10	High Purity	1.0	10	1.0
	ICAL 2	10	High Purity	1.0		1.0
	ICAL 3	1000	High Purity	1.0		100

9.3.3. Matrix Acid Blank (S0)

9.3.3.1. Prepare matrix acid solution by mixing 3% by volume nitric acid and 1% by volume hydrochloric acid in reagent water. Transfer into a clean HDPE bottle and identify the solution as S0.

9.3.3.2. Use this solution for standards or digestate dilutions.

9.3.4. Initial Calibration Standard

9.3.4.1. The initial calibration consists of a blank (S0) and four standards (S1, S2, S3 and S4). Prepare the standards as suggested below. Refer to Table 3 for final concentrations for each analyte. Please note: More standard points may be added at the discretion of the analyst.

Standard Name	ICAL 1 (ml)	ICAL 2 (ml)	Cation Mix (ml)	Trace Mix (ml)	Zn - 10 ppm (ml)	W / Zr – 1 ppm (ml)	P – 100 ppm (ml)	W / Zr – 100 ppm (ml)	Final volume (ml)	Final Concentration (µg/L)
S1	NA	NA	0.050	0.025	0.0025	0.025	0.0125	NA	50	0.5/25/50/1
S2	0.025	0.025	0.500	NA	0.025	0.250	0.025	NA		5/250/500/10/50
S3	0.125	0.125	2.5	NA	0.125	NA	0.125	0.0125		25/1250/2500/50/250
S4	0.250	0.250	5.0	NA	0.250	NA	0.250	0.0250		50/2500/5000/100/500

9.3.5. Continuing Calibration Verification (CCV) Standard

9.3.5.1. Prepare CCV using the stock standards and S0 as suggested below. Refer to Table 3 for final concentrations for each analyte.

Stock Standard	Aliquot (ml)	Final volume (ml)	CCV Final Concentration (µg/ml)
ICAL 1	0.125	50	0.025
ICAL 2	0.125		0.025
Cation Mix	2.500		2.5/1.250
Zn – 10 ppm	0.125		0.050
P	0.125		0.250
Zr	0.0125		0.025
W	0.0125		0.025

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- 9.3.5.2. Prepare intermediate solution for Low Level CCV (LLCCV) using the stock standards and S0 as suggested below. Refer to Table 3 for final concentrations of each analyte.

	Aliquot (ml)	Final volume (ml)	LLCCV Final Concentration (µg/L)
MIX 7A	0.050	50	1.0/100
B – 1 ppm	0.450		9
P – 100 ppm	0.025		50
Sr – 1 ppm	0.050		1
Zn – 10 ppm	0.045		10
Zr – 1 ppm	0.250		5
W – 1 ppm	0.250		5

#### 9.4. Secondary Source Standard

- 9.4.1. Purchase secondary stock standard from a different source as certified standards or equivalent. Refer to list below.

SOURCE	STANDARD	ELEMENTS	CONC. (mg/L)	MATRIX
CPI	EMAX MIX 2	As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, Ti, V, Zn, Ag, Sb, Mo, Li, U, Sn	10	5% HNO <sub>3</sub> + Tr HF
	EMAX MIX 3	Al, Fe, Ca, Mg, K, Na	1000	5% HNO <sub>3</sub>
Phosphorus	CPI	P	1000	0.05% HNO <sub>3</sub>
Zirconium	CPI	Zr	100	2% HNO <sub>3</sub>
Tungsten	Ultra Scientific	W	10	Water

- 9.4.2. Secondary Source Working Standard – Prepare a secondary source working standard using S0 as suggested below or equivalent.

Standard	Element	Stock Conc. (mg/L)	Source	Aliquot (ml)	Final Volume (ml)	Final Conc. (mg/L)
Cation Mix 2	Al	10,000	CPI	2.5	500	50
	Ca	10,000	CPI	2.5		50
	Fe	10,000	CPI	2.5		50
	Mg	10,000	CPI	2.5		50
	K	10,000	CPI	2.5		50
	Na	10,000	CPI	1.25		25

- 9.4.3. Refer to EMAX-QC02 for detailed procedure of standard preparation and labeling.

#### 9.5. Initial Calibration Verification (ICV)

- 9.5.1. Prepare ICV using the secondary stock working standards and S0 as suggested below. Refer to Table 3 for final concentrations for each analyte.

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Stock Standard	Aliquot (ml)	Final Volume (ml)	ICV Final conc. (µg/ml)
ICV1	0.150	50	0.030/0.060
Cation Mix 2	3.000		3.000/1.500
P	0.015		0.300
Zr	0.015		0.030
W	0.015		0.030

9.6. **Interference Standards (ICSA/ICSAB)**

9.6.1. Purchase ICS stock standard as mix certified standards at concentration levels listed below.

STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
6020ICS-0A (ICSA)	Inorganic Ventures	Al, Ca, Fe, Mg, Na, P, K, S	1000	1.4% HNO <sub>3</sub>
		C	2000	
		Cl	10000	
		Mo, Ti	20	
6020ICS-0A + (ICSAB) ICAL 1 + ICAL 2	6020ICS-0A	Al, Ca, Fe, Mg, Na, P, K, S	1000	1.4% HNO <sub>3</sub>
		C	2000	
		Cl	10000	
		Mo, Ti	20	
	ICAL 1	As, B, Se, Tl, Ti, V, Zn, Sb, Mo, Sn	10	5% HNO <sub>3</sub> + Tr HF
	ICAL 2	Ba, Be, Cd, Cr, Co, Cu, Pb, Li, Mn, Ni, Ag, U	10	5% HNO <sub>3</sub> + Tr HF

9.6.2. Prepare Intermediate ICSA and ICSAB standards at concentration levels suggested below. Refer to Table 3 final concentrations.

Standard	Parent Standard	Aliquot (ml)	Final Volume (ml)	Final Concentration (mg/L)
Intermediate ICSA	6020ICS-0A	5	50	Varied
Intermediate ICSAB	6020ICS-0A	5	50	Varied
	ICAL 1	0.10		
	ICAL 2	0.10		
	Zr	0.010		
	W	0.010		

9.7. **LCS/MS Spike Standard**

9.7.1. Purchase LCS/MS standards as certified custom-mixed.

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STANDARD	SOURCE	ELEMENTS	CONC. (mg/L)	MATRIX
ICV 1	CPI	B, Sr, As, Ba, Be, Ag, Cd, Cr, Co, Cu, Ti, Pb, Mn, Ni, Se, V, Zn, Ti, Sb, Mo, Li, U, Sn	10	5% HNO <sub>3</sub> + Tr HF
ICV 2		Al, Fe, Ca, Mg, K, Na	1000	5% HNO <sub>3</sub>
Phosphorus	CPI	P	1000	0.05% HNO <sub>3</sub>
Zirconium	CPI	Zr	100	2% HNO <sub>3</sub>
Tungsten	High Purity	W	10	Water

9.8. **P/A Tuning Standard**

- 9.8.1. Using the calibration stock standard from 9.3.1, prepare a 50 µg/L and 100 µg/L mixed standard. These standards should also include 50 µg/L and 100 µg/L of internal standard, which can be prepared by using the internal standard stock from 9.2.1.

**10.0 PROCEDURES****10.1. Sample Preparation****10.1.1. Water Samples**

- 10.1.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 samples per preparative batch). Withdraw the sample(s) from the sample control room and bring them to the preparation area. Allow the samples to equilibrate at room temperature.
- 10.1.1.2. Shake the sample container. Pour a small amount of sample into the sample cap and trickle just enough to wet the pH indicator strip. Compare the color of the wet strip to the indicator chart displayed in the pH indicator box. Record the pH in the digestion log. If the pH value is <2, proceed to 10.1.3. If the pH value is ≥2, check if special instruction is written on the analysis folder or in the COC. Otherwise, fill out an NCR and inform the supervisor immediately. **DO NOT PROCEED WITH THE DIGESTION. WAIT FOR FURTHER INSTRUCTION.**
- 10.1.1.3. Line up the samples chronologically under the hood. Check and record the lot number of the digestion vessels if it has been verified for accuracy. Take digestion vessels and label each one corresponding to the samples withdrawn and place them in front of each sample making sure that their labels agree. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate.
- 10.1.1.4. Mix the sample thoroughly to achieve homogeneity. Fill each digestion vessel up to the 50-ml mark. (The reduction of the volume is due to waste minimization).
- 10.1.1.5. Record the volume in the digestion log. Use reagent water for blank and LCS.
- 10.1.1.6. Take another digestion vessel; fill it with tap water to 50 ml mark. Put a thermometer inside and let it sit on the digestion block. Turn the thermostat to a pre-determined mark to deliver heat at 90°C - 95°C. Record the temperature reading in the digestion log.
- 10.1.1.7. **Standard Addition**

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10.1.1.7.1. Call for a witness for standard addition. Have the witness verify the setting of the micropipette and the expiration dates of the spike standards.

10.1.1.7.2. Add 0.125 ml of each from EMAX MIX 2 and 3 (see Section 9.4.1) solutions to matrix spike samples and LCS. If Phosphorus, Zirconium and Tungsten are target analytes, add 0.0125 ml of each standard.

**10.1.1.8. Acid Digestion for Dissolved Metals**

10.1.1.8.1. Add 0.5 ml of concentrated HNO<sub>3</sub> and 0.25 ml concentrated HCl to each of the digestion vessels.

10.1.1.8.2. Cap the digestion vessels with disposable watch glass.

10.1.1.8.3. Check that the temperature of the digestion block is ≈ 95°C (covered vessel) and adjust if necessary.

10.1.1.8.4. Place the digestion vessels on the digestion block and reduce volume of sample by continuous heating without boiling for two hours.

10.1.1.8.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion plate and allow the vessels to cool down.

10.1.1.8.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered. Add 1.0 ml concentration HNO<sub>3</sub> and 0.25 ml concentration HCl for matrix matching.

10.1.1.8.7. Dilute the digestate with reagent water to the 50 ml mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new polyethylene container.

**10.1.1.9. Acid Digestion for Total Recoverable Metals**

10.1.1.9.1. Add 0.5 ml of concentrated HNO<sub>3</sub> and 0.25 ml concentrated HCl to each of the digestion vessels.

10.1.1.9.2. Cap the digestion vessels with disposable watch glass.

10.1.1.9.3. Check that the temperature of the digestion block is ≈ 95°C (covered vessel) and adjust if necessary.

10.1.1.9.4. Place the digestion vessels on the digestion block and reduce volume of sample by continuous heating without boiling for two hours.

10.1.1.9.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion block and allow the vessels to cool down.

10.1.1.9.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered. Add 1.0 ml concentration HNO<sub>3</sub> and 0.25 ml concentration HCl for matrix matching.

10.1.1.9.7. Dilute the digestate with reagent water to the 50 ml mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new

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polyethylene container.

**10.1.2. Soil Samples****10.1.2.1. Sample Handling**

10.1.2.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 field samples). Withdraw the sample(s) from the sample control room designated for metals analysis (passing # 10 sieve) and bring them to the weighing area. Allow the samples to equilibrate at room temperature.

*Note: Sample homogeneity is crucial in metals analysis. If sample is not solely designated for metals analysis (i.e., sample is to be used for other analysis) and it is apparent that sample particles contain > #10 sieve, inform the Supervisor for further instruction.*

10.1.2.1.2. Take digestion vessels and label each one corresponding to the samples withdrawn. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate.

10.1.2.1.3. Check project sub-sampling requirement. If multi-incremental sub-sampling (MIS) is required, refer to EMAX-SM01, section 5.13.2 for details. Otherwise follow the steps described in EMAX-SM01, section 5.13.1.

10.1.2.1.4. Scoop 1-2 g sub-sample and transfer into a properly labeled digestion vessel. Record the weight to the nearest 0.01 g.

**10.1.2.2. Pre-heating the Digestion Block**

10.1.2.2.1. Place a digestion vessel with reagent water and a temperature monitoring thermometer on the digestion block.

10.1.2.2.2. Turn the digestion block on and set the thermostat to ~95°C or to a predetermined temperature to obtain approximately 95°C once the digestion vessel is covered with a watch glass.

10.1.2.2.3. When the temperature reading is about 90°-95°C, the digestion block is now ready for digestion.

**10.1.2.3. Standard Addition**

10.1.2.3.1. Call for a witness for standard addition. Have the witness verify the setting of the micropipette and the expiration dates of the spike standards.

10.1.2.3.2. Add 2.5 ml of EMAX MIX 2 & 3 (Sec. 9.4.1) to LCS and matrix spike samples. If Phosphorus and Zirconium are target analytes, add 0.25 ml of each standard. If Tungsten is a target analyte, add 0.025 mL of standard.

**10.1.2.4. Acid Digestion**

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- 10.1.2.4.1. Add 10 ml of reagent water and 5 ml of concentrated HCL<sup>1</sup> into each vessel, swirl the vessel to mix the acid and the sample. Add same amount of acid into a clean and empty vessel and designate it as blank. Insert the vessels in the digestion block(s). Cap the vessels with conical watch glass.
- 10.1.2.4.2. Check the temperature of the digestion block 90°C-95°C, adjust if necessary. If temperature happens to be  $\geq 100^\circ\text{C}$ , adjust the thermostat and wait until temperature falls within 90°C-95°C. Record the temperature reading in the digestion log.
- 10.1.2.4.3. Place the digestion vessels on the digestion block and reflux for 15 minutes without boiling.
- 10.1.2.4.4. Transfer the vessels into unheated digestion block and allow the vessels to cool down for at least 5 minutes. Lift the watch glass and add 10-ml of concentrated HNO<sub>3</sub>. Place the watch glass back before working on the next vessel.
- 10.1.2.4.5. Return the vessels to the digestion block and reflux for another 15 minutes.
- 10.1.2.4.6. Transfer the vessels into unheated digestion blocks and allow the vessels to cool down for at least 5 minutes. Lift the water glass and add 10 ml 1:1 HNO<sub>3</sub>. Place the water glass back before working on the next vessel.
- 10.1.2.4.7. Return the vessels to the digestion block and reflux for another 15 minutes.
- 10.1.2.4.8. Transfer the vessels into unheated digestion blocks and allow the vessels to cool down for at least 5 minutes.
- 10.1.2.4.9. Add 2 ml of reagent water. Then add 3 ml of 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to each vessel, swirling each one of them after every addition to initiate peroxide reaction. Continue to add H<sub>2</sub>O<sub>2</sub> until the amount added reaches 10 ml.
- 10.1.2.4.10. Return the vessels to the heated digestion block. Care must be taken to ensure that losses do not occur due to excessive effervescence.
- 10.1.2.4.11. Continue to reflux the mixture at 90°C-95°C for 15 minutes. Remove the digestion vessels from the digestion block and allow the vessel to cool down for at least 5 mins.
- 10.1.2.4.12. Lift the watch glass, add 5 ml of concentrated HCl. Swirl the vessel until added reagents are properly mixed with the solution. Place the watch glass before working on the next vessel. Return the vessels into the heated digestion block. Reflux for additional 15 minutes. Subsequently, remove the digestion vessels from the digestion block and allow the vessels to cool down and dilute to 100 ml final volume with reagent water.

<sup>1</sup> Addition of 5 ml HCl is a modification from Method 3050B to enhance recovery of antimony. Refer to Appendix 3 for the comparative study done on ICP-MS.

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10.1.2.4.13. Let the digestate settle and centrifuge or filter with Whatman #41 (see 10.1.5) if necessary otherwise digestates are now ready for analysis.

**10.1.2.5. Digestate Filtration**

10.1.2.5.1. Place Whatman #41 filter paper into each funnel resting on holders. Rinse the filter papers with reagent water.

10.1.2.5.2. Place a pre-labeled digestate container under each funnel making sure that the labels are visible.

10.1.2.5.3. Check the labels to make sure that they agree. Pour the digestate into the filter.

10.1.2.5.4. Filter and collect the digestates in the labeled container. The digestates are now ready for analysis.

**10.2. Instrument Parameters**

10.2.1. Set instrument parameters as suggested below.

**10.2.2. Plasma Condition**

- RF Power: 1500 W
- RF Matching: 1.68 V
- Sample Depth: 8.0 mm
- Torch Height: -0.4 mm
- Torch Vertical: 0 mm
- Carrier Gas: 0.9 L/min
- Make-up Gas: 0.15 L/min Note: Total Carrier and Make-up gas not to exceed 1.1 L/min.
- Peristaltic pump: 0.1 rps
- Spray Chamber (S/C) Temp: 2°C

**10.2.3. Ion Lenses**

- Extract 1: 0 V
- Extract 2 : -140V
- Omega Bias-ce: -22 V
- Omega Lens-ce: -1.2 V
- Cell Entrance: -26 V
- QP Focus: 2 V
- Cell Exit: -30 V

**10.2.4. Octopole Parameters**

- Octopole RF: 150 V
- Octopole Bias: -6 V

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- AMU Gain: 127
- AMU Offset: 125
- Axis Gain: 0.9996
- Axis Offset: 0.04
- QP Bias: -3 V

10.2.6. Detector Parameters

- Discriminator: 8 mV
- Analog HV: 1630 V
- Pulse HV: 990 V

10.2.7. Reaction Cell

- H<sub>2</sub> Gas: 3.0 ml/min
- He Gas: 4 ml/min

10.2.8. Adjust the instrument parameters to optimize the instrument performance in conformance to the tuning requirement.

10.2.9. Print the most current instrument parameters and place in the appropriate binder for easy reference. Replaced instrument parameter set-up should be archived chronologically for future reference and historical record.

10.3. **Calibration**10.3.1. Instrument Set-Up

10.3.1.1. Set up the ICP-MS with proper operating parameters. Refer to Section 10.3.

10.3.1.2. Ignite the plasma and allow the instrument to become thermally stable for at least 30 minutes.

10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.2. Tuning the Instrument

10.3.2.1. Tune the instrument according to Normal Mode, Hydrogen Mode and Helium Mode without the internal standard. Refer to Section 10.3 for parameters. On the ICP-MS main Menu, go to Instrument and click Tune and run the tuning solution without the internal standard. After about 60 seconds (making sure the solution is in the system) click start and evaluate the counts of the isotopes according to the table below.

Mode	Range		
Normal	Li6 ≥ 6400 counts	Y89 ≥ 16000 counts	Tl205 ≥ 9600 counts
Hydrogen	Ar/Ar78 < 10	Y89 ≥ 3000 counts	
Helium	V51/Co59 < 0.6	Co59 ≥ 7000 counts	ArCl-75 < 10 counts

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- 10.3.2.2. If non-compliant, adjust parameters (e.g. Torch height, Torch vertical, Octopole Bias and QP Bias) and repeat the tune process until the required range is met.
- 10.3.2.3. Click Generate report for a full scan of the tune. Save all tune values to the current method.
- 10.3.3. Perform a P/A Factor Evaluation
- 10.3.3.1. Analyze the 50 µg/L P/A Tuning Standard.
- 10.3.3.2. Under "Tune," access "P/A Factor."
- 10.3.3.3. Select "Load masses from the acquisition method". Select and delete Ca, (Cd)<sup>106</sup>, (Cd)<sup>108</sup>, (Pb)<sup>206</sup>, (Pb)<sup>207</sup> from the list of elements.
- 10.3.3.4. Select "run."
- 10.3.3.5. When complete accept the changes.
- 10.3.3.6. Print out P/A Factor and store the printout with the tuning data.
- 10.3.3.7. Accept the new P/A Factors.
- 10.3.3.8. Under "file" select "copy tune parameters" and copy the P/A Factors to both the H2 and He modes.
- 10.3.3.9. Save file as "norm.u".
- 10.3.4. Perform Tune Check
- 10.3.4.1. Analyze the intermediate tune check solution (9.1.1) using 4 replicates.
- 10.3.4.2. Evaluate the tune check so that the mass calibration differs no more than 0.1 AMU of the true value and the resolution to be less than 0.9 AMU full width at 10% peak height RSD should less than or equal to 5% for the 4 replicate analysis.
- 10.3.5. Initial Calibration (ICAL)
- 10.3.5.1. Analyze a calibration blank (S0) and a multi-point calibration standard (Section 9.3.3.1).
- 10.3.5.2. Set the instrument rinse time to 90 seconds between each standard solution.
- 10.3.5.3. Refer to Appendix 1 for ICAL acceptance criteria and /or corrective action.
- 10.3.6. Initial Calibration Verification (ICV)/ Instrument Calibration Blank (ICB)
- 10.3.6.1. Analyze the ICV (Section 9.5.1) from a second source to verify the concentration of the ICAL.
- 10.3.6.2. Analyze a low-level ICV (LLICV) from the same source as the calibration standard to verify the lower limit of quantitation (RL).
- 10.3.6.3. Analyze an ICB after LLICV to demonstrate absence of instrument contamination.
- 10.3.6.4. Refer to Appendix 1 for ICV, LLICV and ICB acceptance criteria and /or corrective action.
- 10.3.7. Continuing Calibration Verification (CCV)/ Continuing Calibration Blank (CCB)
- 10.3.7.1. Analyze CCV to check the validity of the ICAL every 10 samples and at the end of the analytical sequence.

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- 10.3.7.2. Analyze low-level CCV (LLCCV) to check the system stability at low end of ICAL at the end of the analytical sequence.
- 10.3.7.3. Analyze a CCB every after LLCCV to demonstrate the absence of instrument contamination.
- 10.3.7.4. Refer to Appendix 1 for CCV, LLCCV and CCB acceptance criteria and/or corrective action.
- 10.3.8. ICSA and ICSAB
- 10.3.8.1. Analyze ICSA and ICSAB at the beginning of each analytical run and every 12 hours thereafter.
- 10.3.9. Establishing Instrument Detection Limit (IDL)
- 10.3.9.1. Analyze a minimum of seven consecutive method blanks.
- 10.3.9.2. Repeat the process within three non-consecutive days.
- 10.3.9.3. Calculate the standard deviation of each run.
- 10.3.9.4. The average of the standard deviation of the three runs determines the IDL for each analyte.
- 10.3.9.5. Establish IDL at least every 3 months.
- 10.3.10. Verifying Linear Dynamic Range (LDR)
- 10.3.10.1. Verify the LDR by preparing a standard at the upper limit of the LDR. Analyze and quantitate against the normal calibration curve. Percent recovery must be within  $\pm 10\%$  of expected value. If non-complaint re-establish LDR.
- 10.3.10.2. At a minimum perform LDR verification every six months.
- 10.4. **Analysis**
- 10.4.1. Analytical Sequence
- 10.4.1.1. From the main menu of the ICPMS top window, go to Sequence and create the analytical sequence by editing the sample log table. Refer to Table 4.
- 10.4.1.2. Set QC limits on QC samples for easy verification while analytical samples are running.
- 10.4.1.3. Using the sample log table, input the standards and the digestates to be analyzed. Samples are analyzed in the order they appear in the scanner.
- 10.4.1.4. Transfer about 5 ml of its content into the autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence. A dilution of x10 for soil samples is required due to the high acid content of the digestate.
- 10.4.1.5. Dilution Test sample is prepared at 5 times dilution. Seal the tube with Parafilm and invert the tube several times to ensure adequate mixing.
- 10.4.1.6. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.
- 10.4.1.7. A 100  $\mu\text{g/L}$  internal standard shall be spiked into each sample.
- 10.4.1.8. Set the prepared analytical samples into the auto-sampler and start the analytical run.

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10.4.2.1. Check QC parameters as soon as the data is available.

- Check the initial calibration verification (ICV, LLICV and ICB) against Appendix 1.
- Check MB, LCS against Appendix 1. Perform specified corrective action if necessary.
- Check the MS, duplicate sample, serial dilution and post digestion spike results. If matrix interference is indicated, dilute the sample and re-analyzed.
- Check intensity of internal standard on each sample.
- If any of the above checkpoints is non-compliant, perform the specified corrective action in Appendix 1. If results indicate digestion problem, order re-digestion for affected samples. If unresolved, consult the Supervisor for further action.

10.4.2.2. Check the sample rack to ensure that the Autosampler did not skip any sample.

10.4.2.3. Check concentration of target analytes. If the response exceeds LDR, dilute and reanalyze the sample at a concentration within the LDR.

10.4.2.4. Check other QC requirements like ICSA, ICSAB, CCV, LLCCV, CCB against Appendix 1.

10.5. **Calculations**

10.5.1. The computer software is designed to calculate the concentration in the digestate, based on the assumption that the initial calibration is linear through the origin. Thus, for aqueous samples, the computer-generated result represent the concentration of the sample.

10.5.2. For water samples

$$C_s = C_i \left( \frac{ExpAmt}{Aliquot} \right) \left( \frac{V_d}{ExpVd} \right) DF \quad \text{Eq.-10.5.2}$$

where:

 $C_s$  – Concentration in the sample, µg/L $C_i$  – Concentration in the digestate, (computer-generated), µg/L $V_d$  – Digestion volume, ml $ExpVd$  – Expected digestion volume, ml $DF$  – Dilution factor $ExpAmt$  – Expected amount for digestion, ml $Aliquot$  – Amount digested, ml10.5.3. For solids, use the following equation to calculate the concentration

$$C_s = C_i \left( \frac{ExpAmt}{Aliquot} \right) \left( \frac{V_d}{ExpVd} \right) \left( \frac{100}{100 - \%H_2O} \right) \times DF \times 0.1 \quad \text{Eq.-10.5.3}$$

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where:

- $C_s$  – Concentration in the sample, mg/Kg  
 $C_i$  – Concentration in the digestate (computer-generated), µg/L  
 $V_d$  – Digestion volume, ml  
 $ExpVd$  – Expected digestion volume, ml  
 $ExpAmt$  – Expected amount for digestion, g  
 $Aliquot$  – Amount digested, g  
 $\% H_2O$  – Percent moisture of the sample  
 $DF$  – Dilution factor  
 $0.1$  – Conversion factor

10.5.4. Calculate the percent recovery (%R)

$$\% Recovery = \frac{C_f - C}{C_s} * 100 \quad \text{Eq.-10.5.4}$$

where:

- $C_f$  – Concentration found, µg/ L  
 $C$  – Concentration of sample, µg/L  
 $C_s$  – Concentration of spike, µg/L

10.5.5. Relative Percent Difference (%RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.5}$$

where:

- $RPD$  – Relative Percent Difference  
 $C_1$  – Measured concentration of the first sample aliquot  
 $C_2$  – Measured concentration of the second sample aliquot

10.6. **Data Reduction**

- 10.6.1. Make a copy of the analytical run log and sample preparation log.  
 10.6.2. Highlight the data to be reported.  
 10.6.3. Print a copy of the raw data and the QC report.  
 10.6.4. Keep all other data generated with the analytical folder marked with “For record only” for traceability purpose.

10.7. **Report Generation**

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- 10.7.1. Print the summary of the analytical run, perform a data transfer into a disk, and convert the instrument electronic output file into a CSV file format.
- 10.7.2. Run the ICCHK.exe program for calibration check.
- 10.7.3. Identify samples that need to be re-analyzed, if any, and report all samples that met the analytical requirements.
- 10.7.4. Generate the report using the following reporting program:

Executable Files	Required Support Files	Output
WDBX <sup>2</sup> .exe	Login File (requires network), project.pln, seq_name.sq, seq_name.ckv, seq_name.ckb, seq_name.csv, seq_name.qck, EXCLCMP.LST	Method.txt [this file integrates the login sample information and the analytical sample information]
IF1VX.exe	method.txt, method.met, method.crf, project.pln, project code.txt, qcell.txt	Sample Results (Form1)
IQCICP.exe	method.txt, method.crf, method.qc, project.pln, project code.txt, qcell.txt	QC Summary for LCS and MS (Form 3)
QCX.exe	method.txt, method.crf, method.qc, project.pln, qcell.txt	Summary for Dilution Test (Form 3)
LABCHRNX.exe	method.txt	Lab Chronicle
CN2.exe	Login File, method.txt, Form 1, Form 3	Case Narrative

**10.7. Data Review**

- 10.8.1. Arrange the analysis package in sequence as detailed below.
- Case Narrative
  - Lab Chronicle
  - Sample Results
  - LCS/LCSD Summary
  - MS/MSD Summary
  - Sample Duplicate Summary
  - Analytical Run Log
  - ICAL Summary
  - ICV Summary
  - CCV Summary

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X<sup>2</sup> – latest program version

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- Sample Preparation Log
  - Non-Conformance Report (if any)
- 10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- Review the ICPCHK.exe output file to ensure that it agrees with the instrument output. Check Project Specific Requirement (PSR) or Appendix 1 for acceptance criteria.
  - Check frequency of calibration verification. Verify results to be within acceptance limits.
  - Check of target analytes concentration to be within linear range.
  - Verify interference check results to be within acceptance limits.
- 10.8.3. If any of the above checkpoints is non-compliant, re-analysis is required.
- 10.8.4. Review the attached logs that they are properly filled.
- 10.8.5. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- 10.8.6. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.
- 10.8.7. Submit the analytical folder for secondary review.
- 10.9. **Preventive Maintenance**
- 10.9.1. Instruments shall receive routine preventive maintenance that is properly recorded in the instrument-specific maintenance logs. The list of maintenance is summarized in Form 6020FM. The practice ensures optimum operating condition of the equipment thus reducing the possibility of frequent instrument malfunction.

Maintenance Activity	Description	Frequency
Verification	Verify instrument parameters to ensure normal operating conditions. Change tubings as necessary. Perform system tune check. Check instrument performance (e.g., ICV/ICB)	Daily prior to analysis
Vacuum System Maintenance	Inspect vacuum hoses and exhaust tubes for possible problems. Check pump for evidence of leakage	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
Ion Lens Cleaning	Remove and clean surfaces of the ion lens. Sonicate ion lens parts	As necessary
System Cleaning	Remove covers and clean dust from fans and vent covers	Every 6 months or as necessary
Pump Maintenance	Replace oil mist filter, drain and replace mechanical pump oil. Verify proper	Every 6 months

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	pump operation	
Inspection	Perform general inspection of the complete system	Once a year

**11.0 QUALITY CONTROL****11.1. Sample Preparation QC**

- 11.1.1. All labwares used in the sample preparation shall be properly treated as specified in EMAX-QC07.
- 11.1.2. A preparative batch consists of 20 or fewer samples of the same matrix that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.
- 11.1.3. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.
- 11.1.4. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

**11.2. Sample Analysis QC**

- 11.2.1. Perform a tune check before every analytical run, an initial calibration and initial calibration verification (ICV / LLICV). Obtain the ICV standard from a different source from that of the initial calibration and LLICV from the same source as the ICAL. Analyze an instrument calibration blank (ICB) after the LLICV. No further analysis shall be valid unless acceptance criteria are met.
- 11.2.2. Monitor the intensities of all internal standards for every analysis. Refer to Appendix 1 for acceptance criteria.
- 11.2.3. Verify inter-element and background correction factors with ICSA and ICSAB standards after ICB every 12 hours.
- 11.2.4. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run. Also verify LLCCV at the end of the analytical run.
- 11.2.5. Evaluate results of MS/MSD to document matrix interference.
- 11.2.6. Perform Post Digestion Spike whenever recoveries for MS/MSD failed.
- 11.2.7. Evaluate Dilution Test result if post digestion spike result failed to meet the acceptance criteria. Failure typically happens when analyte concentrations are high.
- 11.2.8. Refer to Appendix 1 for acceptance criteria.

**11.3. Method QC**

- 11.3.1. A valid DL and LOD must exist prior to sample analysis. Refer to EMAX-QA04 for details.
- 11.3.2. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response unless otherwise specified by the project.
- 11.3.3. All analysts conducting this analysis must have an established Demonstration of Capability (DOC) as described in EMAX-QA05.

**12.0 CORRECTIVE ACTION**

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- 12.1. Quality control procedures and corresponding corrective actions are summarized in Appendix 1.
- 12.2. If tune is non-compliant, consider the following suggestions to correct the problem:
- Check the instrument settings and make sure that the instrument parameters are properly set up.
  - Check argon gas flow.
  - Perform auto tune or visual optimization
  - If the problem persists, inform the Supervisor.
- 12.3. If correlation coefficient (R) of ICAL is non-compliant, consider the following suggestions to help you correct the problem:
- Check the calibration points for possible presence of out-lier. If out-lier is present, prepare a fresh standard and repeat the calibration.
  - Check the connections and make sure that they are air-tight. Perform maintenance as needed.
    - Presence of bubbles is indicative of poor connection between the sipper and the nebulizer.
    - Poor precision to inability to light the plasma is a symptom of a poor drain tube connection
    - Poor precision and carry-over problems are indicative of a dirty spray chamber.
    - Relative increase in the sensitivity ratio of the higher: lower atomic number elements are indicative of stretched pump tubing. The sample flow rate decreases as the tubing stretches.
  - Check the argon gas flow. Loss of signal is indicative of low or no argon gas flow.
  - Poor precision and a gradual loss of signal is indicative of “salting-out” in the nebulizer and/or spray chamber due to samples with high dissolved or suspended solids. This problem will necessitate nebulizer and spray chamber cleaning.
  - If the problem persists, inform the Supervisor.
- 12.4. If ICV is non-compliant, consider the following suggestions to help you correct the problem:
- If the RSD is high it is indicative that the carry-over might be present in the spray chamber.
  - If result is bias high, prepare a fresh standards and repeat calibration.
  - If the problem persists, inform the Supervisor.
- 12.5. If ICB/CCB is non-compliant, consider the following suggestions to correct the problem:
- Prepare a fresh calibration blank solution. Perform instrument rinsing and repeat the ICB/CCB prior to re-analysis of associated sample(s).
  - Carry-over problem is indicative of dirty spray chamber, nebulizer and/or torch. Perform instrument maintenance and repeat the calibration.
  - If the problem persists, inform the Supervisor.
- 12.6. If CCV or LLICV or LLCCV is non-compliant, consider the following suggestions to correct the problem:
- Check the connections prior to re-running the ICAL. Refer to Section 12.3.
  - Prepare a new standard and repeat the ICAL.

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- 12.7. If the intensity of the Internal Standard is non-compliant, consider the following suggestions to correct the problem:
- Check for drift occurrence by observing the internal standard intensities in the calibration blank.
  - If drift has occurred, terminate the analysis, recalibrate, verify the new calibration and reanalyze the affected samples.
  - If drift has not occurred, dilute affected samples five fold and reanalyze with the addition of appropriate amounts of internal standards.
- 12.8. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
- Rule-out instrument contamination by checking the CCBs. Refer to Section 12.5.
  - Rule-out reagent contamination by testing each reagent as described in EMAX-QC01.
  - Rule-out digestate vessel contamination by adding verified reagents heating the vessels prior to testing.
    - Common environmental contaminants – Ca, Si, Fe, Na, Mg, K, Ti, Cu, Mn, can be minimized by maintaining the lab clean.
  - Other sources of contamination:
    - Sweat contains Ca, Mg, Pb, K,  $\text{NH}_4^+$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ , and Cd (for those who smoke).
    - Cosmetics can contain high concentrations of Al, Be, Ca, Cu, Cr, K, Fe, Mn, Ti, and Zn.
    - Some hair dyes contain  $\text{Pb}(\text{OAC})_2$ .
    - Dandruff shampoo can contain significant levels of Se.
    - Eye make-up may contain Hg as a preservative.
    - Calamine lotion is almost pure ZnO.
    - Watches and jewelry contain an assortment of elements and should not be worn in the laboratory.
  - Re-digest MB and the associated samples with reagents free of contamination or with newly opened reagents.
  - If the problem persists, inform the Supervisor.
- 12.9. If LCS is non-compliant, consider the following suggestions to correct the problem:
- If result is bias-high, check the LCS standard by analyzing at the spike level.  
 If the LCS check is within 80-120 % of the expected value, check the calibration of the micropipette use for spiking. Re-digest and re-analyze the LCS and the associated samples.  
 If the LCS check is not within 80-120%, prepare a fresh LCS standard, re-digest and re-analyze LCS and the associated samples.
  - Common Problems with Ag, As, Ba, Pb, and Cr, indicating stock standard degradation, are as follows:
    - Low Silver (Ag) recovery is indicative of Chloride contamination causing  $\text{AgCl}$  precipitation
    - Low Arsenic (As) recovery is indicative of loss during sample preparation as volatile oxides ( $\text{AsO}_3$ ) or precipitation as  $\text{AsCl}_3$
    - Low Barium (Ba) recovery is indicative of  $\text{SO}_4$  or  $\text{CrO}_4$  contamination. Barium will form precipitates with HF and  $\text{H}_2\text{SO}_4$ .

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High Lead (Pb) recovery is indicative of environmental contamination.

12.10. A Non-Conformance Report (NCR) is required when the following circumstances occur:

- Anomalies other than those specified in Appendix 1 are observed.
- Sample is out of technical holding time.

12.10.1. Refer to EMAX-QA08 for NCR details.

**13.0 POLLUTION PREVENTION**

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

**14.0 WASTE MANAGEMENT**

14.1. No sample may be dumped in the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during the analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, digestates, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.1.1 **Preparation Batch** - is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

15.1.1.1 **Analytical batch** - is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.2. Detection Limit (DL) – The lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not a false positive.

15.1.3. Limit of Detection (LOD) – An estimate of the minimum amount of substance that an analytical process can reliably detect.

15.1.4. Limit of Quantitation (LOQ) – The minimum levels, concentrations or quantities of target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

15.1.5. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

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- 15.1.6. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.7. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.8. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.9. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical procedure to monitor contamination.
- 15.1.10. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.11. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.14. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.15. Matrix – is a physical state of a sample. Most of environmental samples are classified as water, soil or air.
- 15.1.16. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.17. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.18. Re-analysis – is a repeated analysis from the same extract/digestate or sample, identified with the Lab Sample ID suffixed with "W".
- 15.1.19. Re-extract/digest – is a repeated sample preparation process identified with the Lab Sample ID suffixed with "R".
- 15.2. **Application of EMAX QC Procedures**
- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing metals analysis. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.
- 15.3. **Department of Defense (DoD) Projects**
- 15.3.1. Samples from DoD sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

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**15.4. Department of Energy (DoE) Projects**

- 15.4.1. Samples from DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, shall be applied.

**16.0 REFERENCES**

- 16.1. "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods", EPA Publication SW-846 Update IV, Method 6020A.
- 16.2. Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry, Method 200.8 Rev. 5.4, 1994.
- 16.3. USEPA SW846, Method 3050B, Revision 2, December 1996.
- 16.4. Title 40 Code of Federal Regulations, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, latest edition.
- 16.5. EMAX Quality Systems Manual, as updated.

**17.0 APPENDICES****17.1. Tables**

- 17.1.1. Table 1 ICP-MS Elements & Isotopes
- 17.1.2. Table 2 Calibration Standard and Verification Preparation
- 17.1.3. Table 3 Calibration Standards Concentration and Reporting Limit
- 17.1.4. Table 4 ICP-MS Analytical Sequence
- 17.1.5. Table 5 DL, LOD, LOQ and Linear Range Concentration Levels

**17.2. Figures**

- 17.2.1. Figure 1 Typical Sample Report
- 17.2.2. Figure 2 Typical LCS/LCD Summary
- 17.2.3. Figure 3 Typical MS/MSD Summary
- 17.2.4. Figure 4 Typical Case Narrative

**17.3. Appendices**

- 17.3.1. Appendix 1 Summary of Quality Control Procedures
- 17.3.2. Appendix 2 Demonstration of Capability
- 17.3.3. Appendix 3 Comparative Study of Modified 3050B

**17.4. Forms**

- 17.4.1. 6020FS Sample Preparation Log
- 17.4.2. 6020FA Analytical Run Log
- 17.4.3. 6020FM Instrument Maintenance Log

**Table 1: ICP-MS ELEMENTS & ISOTOPES**

ELEMENT	SYMBOL	MASS	Tune Mode	Internal Standard
Aluminum	Al	27	3	Sc45
Antimony	Sb	121	3	In115
Arsenic	As	75	2	Ge72
Barium	Ba	137	3	In115
Beryllium	Be	9	3	Li6
Boron	B	11	3	Li6
Cadmium	Cd	111	3	In115
Calcium	Ca	43	1	Sc45
Chromium	Cr	53	2	Sc45
Cobalt	Co	59	3	Sc45
Copper	Cu	63	2	Sc45
Iron	Fe	57	1	Sc45
Lead	Pb	208	3	Tb159
Lithium	Li	7	3	Li6
Magnesium	Mg	24	3	Sc45
Manganese	Mn	55	3	Sc45
Molybdenum	Mo	95	3	In115
Nickel	Ni	60	2	Sc45
Phosphorus	P	31	3	Sc45
Potassium	K	39	3	Sc45
Selenium	Se	78	1	Ge72
Silver	Ag	107	3	In115
Sodium	Na	23	1	Sc45
Strontium	Sr	88	3	Y89
Thallium	Tl	205	3	Tb159
Tin	Sn	118	3	In115
Titanium	Ti	47	3	Sc45
Tungsten	W	182	3	Tb159
Uranium	U	238	3	Tb159
Vanadium	V	50	2	Sc45
Zinc	Zn	66	3	Ge72
Zirconium	Zr	90	3	Ge72

Tune Mode: 1=Reaction H<sub>2</sub> Mode; 2=Collision He Mode; 3= Normal Mode

**Table 2 : CALIBRATION STANDARD AND VERIFICATION PREPARATION**

Standard #	Mixed Standard Name	Conc. (µg/ml)	Source	Preparation		
				Aliquot (ml)	Final Vol. (ml)	Final Conc. (µg/ml)
S1	Trace Mix	1	High Purity	0.025	50	0.0005
	Cation Mix	50/25	AccuStandard	0.050		0.050/0.025
	Zn	10	High Purity	0.0025		0.001
	W	1	AccuStandard	0.025		0.0005
	P	100	AccuStandard	0.0125		0.025
	Zr	1	AccuStandard	0.025		0.0005
S2	ICAL 1	10	High Purity	0.025	50	0.005
	ICAL 2	10	High Purity	0.025		0.005
	Cation Mix	50/25	AccuStandard	0.500		0.500/0.250
	Zn	10	High Purity	0.025		0.010
	W	1	AccuStandard	0.250		0.005
	P	100	AccuStandard	0.025		0.050
S3	Zr	1	AccuStandard	0.250	0.005	
	ICAL 1	10	High Purity	0.125	50	0.025
	ICAL 2	10	High Purity	0.125		0.025
	Cation Mix	50/25	AccuStandard	2.500		2.500/1.250
	Zn	10	High Purity	0.125		0.050
	W	100	AccuStandard	0.0125		0.025
P	100	AccuStandard	0.125	0.250		
S4	Zr	100	AccuStandard	0.0125	0.025	
	ICAL 1	10	High Purity	0.250	50	0.050
	ICAL 2	10	High Purity	0.250		0.050
	Cation Mix	50/25	AccuStandard	5.000		5.000/2.500
	Zn	10	High Purity	0.250		0.100
	W	100	AccuStandard	0.025		0.050
P	100	AccuStandard	0.250	0.500		
ICV	Zr	100	AccuStandard	0.025	0.050	
	ICV 1	10	CPI	0.150	50	0.030/0.060
	Cation Mix 2	50/25	CPI	3.000		3.000/1.500
	W	10	Ultra Scientific	0.150		0.030
	P	1000	CPI	0.015		0.300
Zr	100	CPI	0.015	0.030		
CCV	ICAL 1	10	High Purity	0.125	50	0.025
	ICAL 2	10	High Purity	0.125		0.025
	Cation Mix	50/25	AccuStandard	2.500		2.500/1.250
	W	100	AccuStandard	0.0125		0.025
	Zn	10	High Purity	0.125		0.050
	P	100	AccuStandard	0.125		0.250
LLICV / LLCCV (water)	Zr	100	AccuStandard	0.0125	0.025	
	Mix 7	1/100	High Purity	0.05	50	0.001/0.1
	Zn	10	AccuStandard	0.045		0.01
	W	1	AccuStandard	0.100		0.002
	P	100	AccuStandard	0.025		0.050
Zr	1	AccuStandard	0.25	0.005		
LLICV / LLCCV (soil)	Mix 7	1/100	High Purity	0.025	50	0.0005/0.05
	Zn	10	AccuStandard	0.0025		0.001
	W	1	AccuStandard	0.100		0.002
	P	100	AccuStandard	0.025		0.050
	Zr	1	AccuStandard	0.250		0.005

**Table 3: CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT**

ELEMENT	ICAL (µg/L)				ICV (µg/L)	CCV (µg/L)	ICSA (µg/L)	ICSAB (µg/L)	LLICV,LLCC V, LOQ (Water) (µg/L)	LLICV,LLCC V, LOQ (Soil) (mg/Kg)
	S1	S2	S3	S4						
Aluminum	50	500	2500	5000	3000	2500	100000	100000	100	100
Antimony	0.5	5	25	50	30	25	0	20	1	0.5
Arsenic	0.5	5	25	50	30	25	0	20	1	0.5
Barium	0.5	5	25	50	30	25	0	20	1	0.5
Beryllium	0.5	5	25	50	30	25	0	20	1	0.5
Boron	0.5	5	25	50	30	25	0	200	10	10
Cadmium	0.5	5	25	50	30	25	0	20	1	0.5
Calcium	50	500	2500	5000	3000	2500	100000	100000	100	100
Chromium	0.5	5	25	50	30	25	0	20	1	0.5
Cobalt	0.5	5	25	50	30	25	0	20	1	0.5
Copper	0.5	5	25	50	30	25	0	20	1	0.5
Iron	50	500	2500	5000	3000	2500	100000	100000	100	100
Lead	0.5	5	25	50	30	25	0	20	1	0.5
Lithium	0.5	5	25	50	30	25	0	20	2	0.5
Magnesium	50	500	2500	5000	3000	2500	100000	100000	100	100
Manganese	0.5	5	25	50	30	25	0	20	1	0.5
Molybdenum	0.5	5	25	50	30	25	2000	2000	2	0.5
Nickel	0.5	5	25	50	30	25	0	20	1	0.5
Phosphorus	25	50	250	500	300	250	100000	100000	50	50
Potassium	50	500	2500	5000	3000	2500	100000	100000	100	100
Selenium	0.5	5	25	50	30	25	0	20	1	0.5
Silver	0.5	5	25	50	30	25	0	20	1	0.5
Sodium	25	500	2500	5000	3000	2500	100000	100000	100	100
Strontium	0.5	5	25	50	30	25	0	20	2	0.5
Thallium	0.5	5	25	50	30	25	0	20	1	0.5
Tin	0.5	5	25	50	30	25	0	20	1	20
Titanium	0.5	5	25	50	30	25	2000	2000	2	0.5
Uranium	0.5	5	25	50	30	25	0	20	1	0.5
Vanadium	0.5	5	25	50	30	25	0	20	1	0.5
Zinc	1	10	50	1000	60	50	0	20	20	2
Zirconium	0.5	5	25	50	30	25	0	20	5	5
Tungsten	0.5	5	25	50	30	25	0	20	2	2

**Table 4: ICP-MS ANALYTICAL SEQUENCE**

RUN ID LABEL	SAMPLE DESCRIPTION	SOLUTION ID LABEL
S0	Calibration Standard 1 (blank)	S0
S3, S4, S5	ICAL Standards	S3, S4, S5
ICV	Initial Calibration Verification	ICV
LLICV	Low Level Initial Calibration Verification	LLICV
ICB	Initial Calibration Blank	ICB
ICSA	Initial Interference Solution A	ICSA
ICSAB	Initial Interference Solution A and B	ICSAB
CCV1	Continuing Calibration Verification #1	CCV
CCB1	Continuing Calibration Blank #1	S0
IMSSSSB <sup>3</sup>	Preparation Blank	
IMSSSSL/C	Lab Control Sample	
Sample 1	Sample 1	
Sample 1M	Sample 1 Matrix Spike	
Sample 1S	Sample 1 Matrix Spike Duplicate	
Sample 1J	Sample 1 Serial Dilution(5x dilution sample 1)	
Sample 1A	Sample 1 Post Digestion spike	
Samples 2 to 4	Sample 2 to Sample 5	
CCV2	Continuing Calibration Verification #2	CCV
CCB2	Continuing Calibration Blank #2	S0
Samples 5 to 14	Maximum of 10 Samples	
CCV3	Continuing Calibration Verification #3	CCV
CCB3	Continuing Calibration Blank #3	S0
Samples 15 to 20	Sample 15 to 20 or a maximum of 10 samples (sample 15 to 24)	
ICSA	Initial Interference Solution A	ICSA
ICSAB	Initial Interference Solution B	ICSAB
CCV4	Continuing Calibration Verification #4	CCV
LLCCV	Low Level Continuing Calibration Verification	LLCCV
CCB4	Continuing Calibration Blank #4	S0

<sup>3</sup> where IMSSSS is the digestion batch reference.

**Table 5: DL, LOD, LOQ AND LINEAR RANGE CONCENTRATION LEVELS**

ELEMENT	WATER (µg/L)			SOIL (mg/kg)			LINEAR RANGE µg/L
	DL	LOD	LOQ	DL	LOD	LOQ	
Aluminum	10	20	100	5	10	100	125000
Antimony	0.25	0.5	1	0.1	0.2	0.5	3000
Arsenic	0.1	0.2	1	0.05	0.1	0.5	3000
Barium	0.25	0.5	1	0.072	0.1	0.5	3000
Beryllium	0.05	0.1	1	0.05	0.1	0.5	500
Boron	2.5	5	10	2.5	5	10	250
Cadmium	0.1	0.2	1	0.057	0.1	0.5	3000
Calcium	13	25	100	17	20	100	400000
Chromium	0.1	0.2	1	0.05	0.1	0.5	3000
Cobalt	0.1	0.2	1	0.05	0.1	0.5	3000
Copper	0.25	0.5	1	0.1	0.2	0.5	3000
Iron	5	10	100	5	10	100	300000
Lead	0.05	0.1	1	0.05	0.1	0.5	3000
Lithium	0.25	0.5	2	0.139	0.2	0.5	500
Magnesium	5	10	100	10	20	100	150000
Manganese	0.1	0.2	1	0.153	0.2	0.5	3000
Molybdenum	0.25	0.5	2	0.1	0.2	0.5	3000
Nickel	0.1	0.2	1	0.063	0.1	0.5	3000
Potassium	10	20	100	10	20	100	400000
Phosphorus	12.5	25	50	12.5	25	50	400000
Selenium	0.15	0.3	1	0.05	0.1	0.5	3000
Silver	0.1	0.2	1	0.05	0.1	0.5	250
Sodium	25	50	100	10	20	100	300000
Strontium	0.5	1	2	0.05	0.1	0.5	3000
Thallium	0.1	0.2	1	0.05	0.1	0.5	3000
Tin	0.1	0.2	1	5	10	20	3000
Titanium	0.25	0.5	2	0.125	0.25	0.5	3000
Tungsten	0.5	1	2	0.5	1	2	1000
Uranium	0.05	0.1	1	0.05	0.1	0.5	3000
Vanadium	0.25	0.5	1	0.19	0.3	0.5	3000
Zinc	5	10	20	0.683	1	2	3000
Zirconium	1	2	5	1	2	5	2000

**Figure 1: TYPICAL SAMPLE REPORT**

METHOD 6020A  
 METALS BY ICP-MS

Client	: XYZ, INC.	Date Collected:	11/17/10
Project	: CLEAN PROJECT	Date Received:	11/19/10
SDG NO.	: 10K283	Date Extracted:	11/30/10 17:15
Sample ID:	YWB10-2578	Date Analyzed:	12/03/10 18:05
Lab Samp ID:	K283-10	Dilution Factor:	0.991
Lab File ID:	98L04043	Matrix	: SOIL
Ext Btch ID:	IMK029S	% Moisture	: 11.6
Calib. Ref.:	98L04035	Instrument ID	: EMAXTI98

PARAMETERS	RESULTS (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)
Aluminum	18000	112	22.4
Antimony	ND	0.561	0.112
Arsenic	2.03	0.561	0.112
Barium	51.9	0.561	0.112
Beryllium	0.344J	0.561	0.112
Cadmium	ND	0.336	0.112
Calcium	618	112	22.4
Chromium	5.01	0.561	0.112
Cobalt	3.29	0.561	0.112
Copper	3.12	0.561	0.224
Iron	13300	112	22.4
Lead	4.34	0.561	0.112
Magnesium	2750	112	22.4
Manganese	227	0.561	0.112
Molybdenum	1.24	0.561	0.112
Nickel	3.33	0.561	0.112
Potassium	2050	112	22.4
Selenium	0.188J	0.561	0.112
Silver	ND	0.561	0.112
Sodium	ND	112	22.4
Thallium	0.236J	0.561	0.112
Vanadium	30.0	0.561	0.112
Zinc	28.9	1.12	0.561

**Figure 2: TYPICAL LCS/LCD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN PROJECT  
 SDG NO.: 10K283  
 METHOD: METHOD 6020A

MATRIX: SOIL % MOISTURE: NA  
 DILT N FACTR: 1 1  
 SAMPLE ID: MBLK15  
 CONTROL NO.: IMK0295B IMK0295L IMK0295C  
 LAB FILE ID: 98L04037 98L04038 98L04039  
 DATIME EXTRCTD: 11/30/1017:15 11/30/1017:15 11/30/1017:15 DATE COLLECTED: NA  
 DATIME ANALYZD: 12/03/1017:41 12/03/1017:45 12/03/1017:49 DATE RECEIVED: 11/30/10  
 PREP. BATCH: IMK0295 IMK0295 IMK0295  
 CALIB. REF: 98L04035 98L04035 98L04035

ACCESSION:

PARAMETER	BLNK RSLT mg/kg	SPIKE AMT mg/kg	BS RSLT mg/kg	BS % REC	SPIKE AMT mg/kg	BSD RSLT mg/kg	BSD % REC	RPD %	QC LIMIT %	MAX RPD %
Aluminum	ND	2500	2370	95	2500	2400	96	1	80-120	20
Antimony	ND	25.0	23.7	95	25.0	23.8	95	1	80-120	20
Arsenic	ND	25.0	23.8	95	25.0	23.5	94	1	80-120	20
Barium	ND	25.0	24.2	97	25.0	24.6	98	2	80-120	20
Beryllium	ND	25.0	23.8	95	25.0	23.9	96	1	80-120	20
Cadmium	ND	25.0	23.5	94	25.0	23.8	95	1	80-120	20
Calcium	ND	2500	2490	99	2500	2490	99	0	80-120	20
Chromium	ND	25.0	24.4	98	25.0	24.7	99	1	80-120	20
Cobalt	ND	25.0	23.8	95	25.0	24.1	96	1	80-120	20
Copper	ND	25.0	24.2	97	25.0	24.4	98	1	80-120	20
Iron	ND	2500	2440	98	2500	2450	98	0	80-120	20
Lead	ND	25.0	24.5	98	25.0	24.4	97	0	80-120	20
Magnesium	ND	2500	2370	95	2500	2390	95	1	80-120	20
Manganese	ND	25.0	23.8	95	25.0	24.0	96	1	80-120	20
Molybdenum	ND	25.0	24.2	97	25.0	24.2	97	0	80-120	20
Nickel	ND	25.0	24.3	97	25.0	24.6	98	1	80-120	20
Potassium	ND	2500	2440	98	2500	2450	98	0	80-120	20
Selenium	ND	25.0	23.8	95	25.0	23.9	95	0	80-120	20
Silver	ND	25.0	23.6	94	25.0	23.9	96	1	80-120	20
Sodium	ND	2500	2460	98	2500	2440	98	1	80-120	20
Thallium	ND	25.0	23.8	95	25.0	24.0	96	1	80-120	20
Vanadium	ND	25.0	24.3	97	25.0	24.5	98	1	80-120	20
Zinc	ND	25.0	25.0	100	25.0	24.7	99	1	80-120	20

**Figure 3: TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN PROJECT  
 SDG NO.: 10K283  
 METHOD: METHOD 6020A

MATRIX: SOIL % MOISTURE: 11.6  
 DILTN FACTR: 0.991 0.992 0.992  
 SAMPLE ID: YWB10-2578  
 CONTROL NO.: K283-10 K283-10M K283-10S  
 LAB FILE ID: 98L04043 98L04040 98L04041  
 DATIME EXTRACTD: 11/30/1017:15 11/30/1017:15 11/30/1017:15 DATE COLLECTED: 11/17/10  
 DATIME ANALYZD: 12/03/1018:05 12/03/1017:53 12/03/1017:57 DATE RECEIVED: 11/19/10  
 PREP. BATCH: IMK029S IMK029S IMK029S  
 CALIB. REF: 98L04035 98L04035 98L04035

ACCESSION:

PARAMETER	SMPL RSLT mg/kg	SPIKE AMT mg/kg	MS RSLT mg/kg	MS % REC	SPIKE AMT mg/kg	MSD RSLT mg/kg	MSD % REC	RPD %	QC LIMIT %	MAX RPD %
Aluminum	18000	2810	21900	139*	2810	23200	187*	6	80-120	20
Antimony	ND	28.1	23.5	84	28.1	24.5	87	4	80-120	20
Arsenic	2.03	28.1	26.7	88	28.1	27.9	92	5	80-120	20
Barium	51.9	28.1	84.3	116	28.1	90.1	136*	7	80-120	20
Beryllium	0.344J	28.1	26.5	93	28.1	27.0	95	2	80-120	20
Cadmium	ND	28.1	25.8	92	28.1	26.8	95	3	80-120	20
Calcium	618	2810	3330	97	2810	3360	98	1	80-120	20
Chromium	5.01	28.1	31.4	94	28.1	33.1	100	5	80-120	20
Cobalt	3.29	28.1	28.5	90	28.1	29.5	93	3	80-120	20
Copper	3.12	28.1	28.6	91	28.1	29.7	95	4	80-120	20
Iron	13300	2810	17000	131*	2810	18300	176*	7	80-120	20
Lead	4.34	28.1	30.8	94	28.1	32.6	101	6	80-120	20
Magnesium	2750	2810	5550	100	2810	5770	108	4	80-120	20
Manganese	227	28.1	274	170*	28.1	285	207*	4	80-120	20
Molybdenum	1.24	28.1	27.2	93	28.1	28.5	97	5	80-120	20
Nickel	3.33	28.1	29.3	92	28.1	30.7	98	5	80-120	20
Potassium	2050	2810	5000	105	2810	5170	111	3	80-120	20
Selenium	0.188J	28.1	24.4	86	28.1	25.1	89	3	80-120	20
Silver	ND	28.1	26.1	93	28.1	27.0	96	3	80-120	20
Sodium	ND	2810	2620	93	2810	2680	96	2	80-120	20
Thallium	0.236J	28.1	25.9	92	28.1	27.3	96	5	80-120	20
Vanadium	30.0	28.1	58.0	100	28.1	63.4	119	9	80-120	20
Zinc	28.9	28.1	57.7	103	28.1	60.5	113	5	80-120	20

**Figure 4: TYPICAL CASE NARRATIVE**

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN PROJECT  
SDG : 10K283

METHOD 6020A  
METALS BY ICP-MS

A total of twelve (12) soil samples were received on 11/19/10 for Total Metals by ICP-MS analysis, Method 6020A in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Initial Calibration was established as prescribed by the method and was verified using a secondary source. Interference checks were performed and results were within required limits. Continuing calibration verifications and continuing calibration blanks were carried out at the frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for IMK029SL/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at the frequency prescribed by the project. Percent recoveries were within project QC limits except for results qualified with [\*] in K283-10M/S summary form, most likely due to matrix interference. Check QC summaries form for details. In addition Analytical spike and serial dilution were analyzed for matrix interference evaluation. Results were within method acceptance criteria.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

## Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURES	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Tune Check (Mass calibration and resolution check)	Daily before ICAL.	±0.10 AMU (Mass of Isotope) <0.9 AMU full width resolution RSD of 4 replicates : ≤5%	Correct problem and repeat tune check.		
Initial Calibration (multi-point)	Daily initial calibration prior to sample analysis.	$r \geq 0.998$	Correct the problem and repeat the initial calibration.		
Initial Calibration Verifications (ICV) Second Source	Daily after the initial calibration.	All analytes within ±10% of expected value RSD of Replicate integrations: < 5%	Correct the problem and repeat the initial calibration.		
Low Level Calibration Verification (LLICV / LLCCV)	LLICV: Daily after initial calibration. LLCCV: At the end of the analysis sequence	All analytes with ± 30% of expected value.	Correct the problem and repeat the initial calibration.		
Calibration Verifications (CCV)	Daily before sample analysis, after every 10 samples and at the end of the analysis sequence.	All analytes within ±10% of expected value. RSD of replicate integrations < 5%.	Repeat calibration and reanalyze all samples since last successful calibration.		
Calibration Blanks (ICB/CCB)	After every calibration verification	All target analytes < LOQ.	Correct problem then reanalyze calibration blank and previous samples.		
Interference Check Sample (ICSA/ICSAB)	Analyze at the beginning of each analytical run or once every 12 hours, whichever is more frequent.	Within ±20% of expected value	Terminate analysis, correct the problem, reanalyze ICS, and reanalyze all affected samples		
Internal Standard (IS)	ICV, LLICV, CCV, LLCCV, CCBs, MB, LCS, every sample	IS Intensities > 70% from Initial Calibration Blank IS Intensity	Correct problem then re-analyze		
Method Blank	One per preparation batch	All target analytes < ½ LOQ.	Re-digest and reanalyze method blank and all samples processed with the contaminated blank.		
Laboratory Control Sample (LCS)	One per preparation batch	% Recovery: 80% - 120%	Re-digest and reanalyze LCS and all associated samples		
Matrix Spikes (MS/MSD)	One MS/MSD every 20 project samples per matrix	% Recovery: 75% - 125% RPD ≤20%	Evaluate post spike and dilution test: <ul style="list-style-type: none"> <li>If parent sample result is "ND", evaluate post spike.</li> <li>If parent sample result is high (i.e., 4x of spike concentration) and post spike failed, evaluate dilution test.</li> </ul>		
Post Digestion Spike Addition	When MS fails.	Recovery within 80-120% of expected value	Correct the problem then reanalyze post digestion spike addition		
Dilution Test (5X)	When MS fails.	1:5 dilution must agree within ±10% of the original determination	Evaluate. Discuss in case narrative.		
Instrument Detection Limit (IDL)	Every three months		Correct the problem and repeat the IDL determination.		
Comments: Refer to PSR for flagging criteria.				Reviewed By:	
				Date:	

**Appendix 2: DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: SW 6020A**

SOP: EMAX-6020  
Conc Unit: µg/L  
Sample Amt(ml): 50

Instrument ID: I98  
Analysis date: 1/7/2011  
Analyzed by: C. Capulong

**MATRIX : WATER**

PARAMETER	98A04019	98A04020	98A04021	98A04022	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	IMA006WL	IMA006WC	IMA006WX	IMA006WY							
Aluminum	2513	2514	2516	2513	2500	2514	101	1.41	0.06	80 - 120	PASSED
Antimony	25.1	25.1	25.2	25.0	25	25	100	0.09	0.36	80 - 120	PASSED
Arsenic	24.6	25.0	24.8	24.8	25	25	99	0.17	0.67	80 - 120	PASSED
Barium	25.7	25.6	25.7	25.4	25	26	102	0.15	0.58	80 - 120	PASSED
Beryllium	26.2	26.4	26.0	26.4	25	26	105	0.18	0.67	80 - 120	PASSED
Boron	27.3	27.8	27.2	27.7	25	28	110	0.31	1.13	80 - 120	PASSED
Cadmium	25.4	25.3	25.5	25.2	25	25	101	0.13	0.50	80 - 120	PASSED
Calcium	2578	2555	2563	2585	2500	2570	103	13.70	0.53	80 - 120	PASSED
Chromium	25.4	25.4	25.3	25.0	25	25	101	0.18	0.72	80 - 120	PASSED
Cobalt	25.4	26.0	25.6	25.6	25	26	103	0.22	0.87	80 - 120	PASSED
Copper	25.6	25.8	25.7	25.3	25	26	102	0.22	0.86	80 - 120	PASSED
Iron	2600	2583	2596	2594	2500	2593	104	7.27	0.28	80 - 120	PASSED
Lead	25.7	25.8	25.5	25.6	25	26	103	0.10	0.40	80 - 120	PASSED
Lithium	24.6	24.9	24.7	25.1	25	25	99	0.22	0.88	80 - 120	PASSED
Magnesium	2510	2516	2492	2506	2500	2506	100	10.20	0.41	80 - 120	PASSED
Manganese	25.6	25.7	25.7	25.4	25	26	102	0.15	0.59	80 - 120	PASSED
Molybdenum	25.1	25.1	25.1	24.7	25	25	100	0.19	0.74	80 - 120	PASSED
Nickel	25.3	25.6	25.4	25.0	25	25	101	0.23	0.91	80 - 120	PASSED
Potassium	2602	2593	2602	2558	2500	2589	104	20.93	0.81	80 - 120	PASSED
Selenium	25.4	25.1	25.3	25.0	25	25	101	0.16	0.62	80 - 120	PASSED
Silver	25.4	25.6	25.5	25.0	25	25	101	0.27	1.05	80 - 120	PASSED
Sodium	2507	2502	2507	2544	2500	2515	101	19.48	0.77	80 - 120	PASSED
Strontium	25.3	25.4	25.3	25.2	25	25	101	0.09	0.35	80 - 120	PASSED
Thallium	25.5	25.9	25.7	25.7	25	26	103	0.16	0.63	80 - 120	PASSED
Tin	25.1	25.2	25.5	25.3	25	25	101	0.15	0.59	80 - 120	PASSED
Titanium	25.2	25.0	25.2	24.8	25	25	100	0.17	0.66	80 - 120	PASSED
Uranium	26.1	26.1	25.9	25.9	25	26	104	0.13	0.50	80 - 120	PASSED
Vanadium	25.3	25.4	25.2	24.8	25	25	101	0.26	1.03	80 - 120	PASSED
Zinc	25.3	25.4	25.6	25.2	25	25	101	0.15	0.59	80 - 120	PASSED

**Appendix 2 (Cont.): DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: SW 6020A**

Analytical SOP: EMAX-6020  
Conc Unit: µg/L  
Sample Amt(m): 50

Analysis date: 5/9/2011  
Extracted by: C. Capulong  
Analyzed by: C. Capulong

PARAMETER	98E05041	98E05042	98E05043	98E05044	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	IME016WL	IME016WC	IME016WX	IME016WY							
Phosphorus	236.9	244.8	236.5	237.6	250	239	96	3.93	1.64	80 - 120	PASSED
Zirconium	25.0	27.2	25.8	26.3	25	26.1	104	0.93	3.55	80 - 120	PASSED

**DEMONSTRATION OF CAPABILITY  
METHOD: SW 6020A**

SOP: EMAX-6020  
Conc Unit: µg/L  
Sample Amount(mL): 50

Instrument ID: 98  
Analysis date: 6/25/2013  
Analyzed by: C. Capulong

PARAMETER	98F06024	98F06025	98F06026	98F06027	TV	Ave. Conc.	Ave. %Rec	SD	RSD	QC Criteria	COMMENTS
	IMF012WL	IMF012WC	IMF012WX	IMF012WY							
Tungsten	26.8	26.7	26.4	26.6	25	26.3	105	0.15	0.6	80 - 120	PASSED

**Appendix 2 (Cont.): DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: SW 6020A**

SOP: EMAX-6020  
Conc Unit: mg/Kg  
Sample Amt(gm): 1

Instrument ID: I98  
Analysis date: 1/7/2011  
Analyzed by: C. Capulong

**MATRIX : SOIL**

PARAMETER	98A04027	98A04028	98A04029	98A04030	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	IMA007SL	IMA007SC	IMA007SX	IMA007SY							
Aluminum	2439	2397	2422	2435	2500	2423	97	18.95	0.78	80 - 120	PASSED
Antimony	21.1	20.9	20.8	20.8	25	21	84	0.13	0.61	80 - 120	PASSED
Arsenic	20.7	20.5	20.9	20.9	25	21	83	0.22	1.04	80 - 120	PASSED
Barium	21.6	21.5	21.4	21.7	25	22	86	0.12	0.54	80 - 120	PASSED
Beryllium	21.7	21.7	21.7	21.6	25	22	87	0.05	0.23	80 - 120	PASSED
Boron	23.0	23.2	23.4	23.5	25	23	93	0.23	1.00	80 - 120	PASSED
Cadmium	21.1	20.8	20.7	21.0	25	21	83	0.19	0.92	80 - 120	PASSED
Calcium	2535	2511	2502	2524	2500	2518	101	14.49	0.58	80 - 120	PASSED
Chromium	21.3	21.3	21.5	21.2	25	21	85	0.11	0.51	80 - 120	PASSED
Cobalt	21.5	21.2	21.6	21.5	25	21	86	0.20	0.95	80 - 120	PASSED
Copper	21.1	21.2	21.2	20.8	25	21	84	0.17	0.82	80 - 120	PASSED
Iron	2509	2501	2470	2525	2500	2501	100	23.10	0.92	80 - 120	PASSED
Lead	21.8	21.6	21.5	21.6	25	22	87	0.13	0.59	80 - 120	PASSED
Lithium	20.9	21.1	21.1	21.1	25	21	84	0.07	0.31	80 - 120	PASSED
Magnesium	2424	2401	2413	2427	2500	2416	97	11.81	0.49	80 - 120	PASSED
Manganese	21.5	21.5	21.6	21.5	25	22	86	0.06	0.29	80 - 120	PASSED
Molybdenum	21.4	21.2	21.3	21.4	25	21	85	0.11	0.53	80 - 120	PASSED
Nickel	20.9	20.8	21.0	20.8	25	21	83	0.08	0.36	80 - 120	PASSED
Potassium	2493	2499	2495	2483	2500	2493	100	6.81	0.27	80 - 120	PASSED
Selenium	21.1	20.8	21.3	21.1	25	21	84	0.19	0.92	80 - 120	PASSED
Silver	21.6	21.4	21.5	21.8	25	22	86	0.17	0.80	80 - 120	PASSED
Sodium	2440	2435	2390	2464	2500	2432	97	30.88	1.27	80 - 120	PASSED
Strontium	21.5	21.3	21.5	21.7	25	21	86	0.17	0.80	80 - 120	PASSED
Thallium	21.6	21.4	21.4	21.5	25	21	86	0.08	0.39	80 - 120	PASSED
Tin	24.6	24.2	24.1	24.3	25	24	97	0.24	1.00	80 - 120	PASSED
Titanium	21.6	21.4	21.5	21.5	25	22	86	0.08	0.38	80 - 120	PASSED
Uranium	22.4	22.0	22.4	22.4	25	22	89	0.21	0.96	80 - 120	PASSED
Vanadium	21.4	21.2	21.3	21.1	25	21	85	0.12	0.57	80 - 120	PASSED
Zinc	22.6	21.8	21.9	22.7	25	22	89	0.47	2.11	80 - 120	PASSED

**Appendix 2 (Cont.): DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
 METHOD: SW 6020A**

Analytical SOP: EMAX-6020  
 Conc Unit: mg/Kg  
 Sample Amt(ml): 1

Analysis date: 5/9/2011  
 Extracted by: C. Capulong  
 Analyzed by: C. Capulong

PARAMETER	98E05021	98E05022	98E05023	98E05024	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	IME012SL	IME012SC	IME012SX	IME012SY							
Phosphorus	257	255	255	252	250	255	102	2.25	0.88	80 - 120	PASSED
Zirconium	27.3	29.3	29.7	29.5	25	28.9	116	1.12	3.87	80 - 120	PASSED

**DEMONSTRATION OF CAPABILITY  
 METHOD: SW 6020A**

SOP: EMAX-6020  
 Conc Unit: mg/Kg  
 Sample Amount(g): 1

Instrument ID: 98  
 Analysis date: 6/25/2013  
 Analyzed by: C. Capulong

PARAMETER	98F06019	98F06020	98F06021	98F06022	TV	Ave. Conc.	Ave. %Rec	SD	RSD	QC Criteria	COMMENTS
	IMF014SL	IMF014SC	IMF014SX	IMF014SY							
Tungsten	25.8	25.9	26.1	26.1	25	25.8	103	0.15	0.6	80 - 120	PASSED

**Appendix 3:**

**COMPARATIVE STUDY OF MODIFIED 3050B**

ANALYTICAL METHOD: SW 6020A  
 PREPARATION BATCH: IMG031S - Modified Method 3050B  
 IMG032S - Reference Method 3050B  
 ANALYTICAL BATCH: I98H01  
 QC STANDARD: 09G281-02

Preparation Date: 7/27/2010  
 Extracted by: M. Mendoza  
 Analytical Run Date: 8/3/2010  
 Analyzed by: C. Capulong

COMPOUND	TRUE VALUES (mg/Kg)	ACCEPTANCE LIMITS (mg/Kg)	RECOVERY LIMITS (%)	REFERENCE METHOD 3050B				MODIFIED METHOD 3050B			
				Concentration (mg/Kg)		Recovery		Concentration (mg/Kg)		Recovery	
				98H01041 G281-02	98H01043 G281-02D	98H01041 G281-02	98H01043 G281-02D	98H01030 G281-02	98H01032 G281-02D	98H01030 G281-02	98H01032 G281-02D
Aluminum	7320	4940 - 16400	67 - 224	10900	10700	149%	146%	10700	10700	146%	146%
Antimony	110	11.0 - 121	10 - 110	20.0	19	18%	17%	61.4	66.6	56%	61%
Arsenic	84.2	42.8 - 92.7	51 - 110	61.2	61.7	73%	73%	63.6	65.3	76%	78%
Barium	247	186 - 318	75 - 129	255	260	103%	105%	275	273	111%	111%
Beryllium	49.0	29.0 - 53.9	59 - 110	39.7	39.6	81%	81%	42	41.7	86%	85%
Boron	130	61.8 - 149	48 - 115	104	104	80%	80%	126	126	97%	97%
Cadmium	76.5	48.7 - 84.4	64 - 110	65.3	66.7	85%	87%	69.9	69.8	91%	91%
Calcium	10200	7860 - 12900	77 - 126	9360	10700	92%	105%	10200	10300	100%	101%
Chromium	116	74.8 - 140	64 - 121	99.5	98.4	86%	85%	107	110	92%	95%
Cobalt	64.6	47.2 - 79.7	73 - 123	62.1	61.3	96%	95%	63.8	64	99%	99%
Copper	143	99.7 - 166	70 - 116	116	115	81%	80%	127	126	89%	88%
Iron	23300	11500 - 36200	49 - 155	23900	23700	103%	102%	24800	24800	106%	106%
Lead	82.0	41.9 - 90.2	51 - 110	63.3	64.9	77%	79%	67.5	67.6	82%	82%
Magnesium	7400	5300 - 9060	72 - 122	6770	6760	91%	91%	6930	7100	94%	96%
Manganese	475	428 - 724	90 - 152	546	562	115%	118%	584	571	123%	120%
Molybdenum	66.1	36.0 - 72.7	54 - 110	54.9	53.5	83%	81%	60.1	59.4	91%	90%
Nickel	144	88.8 - 158	62 - 110	112	113	78%	78%	122	122	85%	85%
Potassium	3740	2360 - 4990	63 - 133	3620	3600	97%	96%	3690	3560	99%	95%
Selenium	189	97.2 - 208	51 - 110	142	148	75%	78%	154	157	81%	83%
Silver	40.6	21.2 - 44.6	52 - 110	30.9	32.7	76%	81%	34.2	34.9	84%	86%
Sodium	200	31.4 - 324	16 - 162	292	197	146%	99%	192	200	96%	100%
Strontium	135	88.1 - 162	65 - 120	123	121	91%	90%	129	128	96%	95%
Thallium	127	73.2 - 144	58 - 113	110	114	87%	90%	117	112	92%	88%
Tin	250	39.5 - 275	16 - 110	71.2	73.2	28%	29%	84.7	85.2	34%	34%
Titanium	609	426 - 792	70 - 130	737	744	121%	122%	772	760	127%	125%
Uranium	1.12	0.784 - 1.46	70 - 130	0.93	1.1	83%	98%	1.03	1.01	92%	90%
Vanadium	61.8	55.6 - 123	90 - 199	90.2	89.7	146%	145%	92.8	93.1	150%	151%
Zinc	208	145 - 270	70 - 130	208	207	100%	100%	225	214	108%	103%

\* Note: Modified 3050B procedure is specified in EMAX-3050 Rev. 4







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SOP REVIEW FORM

EMAX-7199

SOP No.

Rev. 3

Revision Number

HEXAVALENT CHROMIUM

Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all MSDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation for Aqueous Sample	/			
- Sample Preparation for Soil Sample	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			Update Sop to include 3060 requirements

SOP update can be done as an addendum.

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

*Farina Madamba*  
 FARINA MADAMBA

Date:

2/11/15

STANDARD OPERATING PROCEDURES  
**HEXAVALENT CHROMIUM**

SOP No.: EMAX-7199 Revision No. 3 Date: 20-Nov-12  
 Prepared By: Lucita Arzadon *L.A. Arzadon* Date: 11/20-12  
 Approved By: Kenette Pimentel *K. Pimentel* Date: 11-20-12  
 QA Manager  
 Approved By: Caspar Pang *C. Pang* Date: 11-20-12  
 Laboratory Director

Control Number: **7199-03-**

### 1.0 **SCOPE AND APPLICATION**

- 1.1. This method is applicable for the determination of hexavalent chromium in: ground waters, reagent waters, by Ion Chromatography.
- 1.2. This SOP is an adaptation of USEPA Method 7199.

### 2.0 **SUMMARY OF METHOD**

- 2.1. An aqueous sample is filtered through a 0.45 um filter and the filtrate is adjusted to a pH of 9.0 to 9.5 with a buffer solution. A measured volume of the sample (250-1000uL) is introduced into the ion chromatograph. A guard column removes organics from the sample before the Cr(VI) as  $\text{CrO}_4^{2-}$  is separated on an anion exchange separator column. Post-column derivatization of the Cr(VI) with diphenylcarbazide is followed by detection of the colored complex at 530nm.
- 2.2. **Interferences**
  - 2.2.1. **Contamination** – A trace amount of Cr is sometimes found in reagent grade salts. Since a concentrated buffer solution is used in this method to adjust the pH of samples, reagent blanks should be analyzed to assess for potential Cr(VI) contamination. Contaminations can also come from improperly cleaned glassware or contact or caustic or acidic reagents of samples with stainless steel or pigmented material.
  - 2.2.2. Reduction of Cr(VI) to Cr(III) can occur in the presence of reducing species in an acidic medium. However, at a pH of 6.5 or greater,  $\text{CrO}_4^{2-}$ , which is less reactive than the  $\text{HCrO}_4^-$ , is the predominant species.
  - 2.2.3. Overloading of the analytical column capacity with high concentrations of anionic species, especially chloride and sulfate, will cause a loss of Cr(VI). The column specified in this method can handle samples containing up to 5% sodium sulfate or 2% sodium chloride. Poor recoveries from fortified samples and tailing peaks are typical manifestations of column overload.

### 3.0 **DETECTION LIMITS**

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**
  - 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
  - 3.1.2. Established DL, LOD and LOQ are:

MATRIX	DL	LOD	LOQ
Water (Regular Level), µg/L	0.05	0.1	0.20

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

SOP No.: EMAX-7199 Revision No. 3 Date: 20-Nov-12

MATRIX	DL	LOD	LOQ
Water (Low Level), µg/L	0.0125	0.025	0.05
Soil (Alkali Digestion), µg/Kg	13	20	40
Soil (Leaching), µg/Kg	0.5	1	2

**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.3). All samples analyzed above this range shall be considered “over range” and shall require dilution to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range shall be considered as “under range” and shall require lower dilution factor to properly quantitate.

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1. Aqueous samples**

- 5.1.1. Aqueous samples are expected to be received in either HPDE or glass bottles cooled at  $\leq 6^{\circ}\text{C}$  after collection.
- 5.1.2. The holding time is 24 hours from the time of sample collection. Store samples at  $\leq 6^{\circ}\text{C}$  without freezing until analysis is completed.
- 5.1.3. Samples may be preserved with Ammonium Hydroxide – Ammonium Sulfate buffer solution to extend holding time to 28 days.

**5.2. Soil samples**

- 5.2.1. Soil samples are expected to be received in glass jars cooled at  $\leq 6^{\circ}\text{C}$  after collection.
- 5.2.2. Extract samples within 30 days from sampling date. Store samples at  $\leq 6^{\circ}\text{C}$  until analysis is completed.
- 5.2.3. For samples extracted by leaching, extracts must be analyzed within 24 hours of leaching. For alkali digestion, extracts must be analyzed within 168 hours after digestion.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA04 Detection Limit (DL)
- 6.3. EMAX-QC02 Analytical Standard Preparation
- 6.4. EMAX-SM04 Analytical and QC Sample Labeling
- 6.5. EMAX-3060 Alkaline Digestion for Hexavalent Chromium

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

SOP No.: EMAX-7199 Revision No. 3 Date: 20-Nov-12

**7.0 SAFETY**

- 7.1. Read all MSDS for chemicals listed in this SOP.
- 7.2. Treat reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. If, for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS & REAGENTS****8.1. Instruments****8.1.1. Ion Chromatography**

Instrument ID	Inst. 59	Inst. D6	Inst. G2
IC	Dionex LC20/AD25	Dionex ICS-1000 /AD25	Metrohm Compact IC Pro881
Guard Column	Dionex NGI	Dionex NGI	Metrosep RP Guard
Column	Dionex Ion Pack AS7	Dionex Ion Pack AS7	Metrosep A Supp7
Detector	UV/VIS AD25 Absorbance Detector	UV/VIS AD25 Absorbance Detector	Metrohm Professional UV/VIS Detector 887
Autosampler	AS40 Automated Autosampler	AS50 Autosampler	Metrohm Professional Sample Collector 858
Data Acquisition	Chromeleon 6.7V	Chromeleon 6.7V	Magic Net 2.3

- 8.1.2. Balance – Top Loading, sensitivity  $\pm 0.0001$  g
- 8.1.3. pH Meter
- 8.1.4. Digester (for Alkaline Digestion) – capable of heating at 90-95°C
- 8.1.5. Thermometer – range 0-110°C

**8.2. Chemicals**

Instrument ID	Inst. 59 and D6	Inst. G2
Reagent water	Deionized water (ASTM Type II) or equivalent	Deionized water (ASTM Type II) or equivalent
Eluent solution	Dissolve 66g of ammonium sulfate in 1L reagent water; add 13mL ammonium hydroxide. Dilute to 2L with reagent water.	12.8 mM Na <sub>2</sub> CO <sub>3</sub> / 4.0mM NaHCO <sub>3</sub> Add 80mL of 320mM Na <sub>2</sub> CO <sub>3</sub> / 100mM NaHCO <sub>3</sub> into a 2L volumetric flask and dilute to the mark with reagent water.
Buffer Solution	Dissolve 165g of (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> into 250mL of H <sub>2</sub> O and add 32.5mL NH <sub>4</sub> OH. Dilute to	Dissolve 3.3g of ammonium sulfate (Assay 99.0%) in 75mL reagent water; add 6.5mL

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

SOP No.: EMAX-7199

Revision No. 3

Date: 20-Nov-12

Instrument ID	Inst. 59 and D6	Inst. G2
	500mL with reagent water.	ammonium hydroxide (Assay 20-22%). Dilute to 100mL with reagent water.
Post Column Reagent	Add 28mL of 98% sulfuric acid to 500-mL of reagent water in a 1L flask. In a separate container (125mL plastic snap seal), dissolve 0.5g of 1,5-diphenyl carbazide in 100mL of HPLC-grade methanol. Once dissolved, add the solution to the flask. Dilute to 1L with reagent water.	Add 28mL of 98% sulfuric acid to 500-mL of reagent water in a 1L flask. In a separate container (125mL plastic snap seal), dissolve 0.5g of 1,5-diphenyl carbazide in 100mL of HPLC-grade methanol. Sonicate for five (5) minutes to dissolve. Once dissolved, add the solution to the flask. Dilute to 1L with reagent water.
Gas	Nitrogen gas, high purity grade	Not applicable

**Reagents**

NaOH (2.5%) – for pH adjustment

Silica Sand – for soil blank

**8.3. Supplies**

Autosampler vials	5mL with filter caps(Inst. 59); 10mL polyvial vials (Inst D6); 11 mL PP sample tubes (Inst. G2)
Filters	0.45µm Phenomenex and 0.20 µm Phenomenex or equivalent
Volumetric Flasks	100, 200, 250, 1000 mL
Containers	40 mL and 125 mL polyethylene snap seal
Micropipettes	1 and 5 mL; 2 and 200 µL
Graduated Cylinder	100 -mL class A
pH Strip/pH meter	Narrow 7-14 @ 0.5 graduation

**9.0 STANDARDS****9.1. Standard Preparation**

9.1.1. Refer to EMAX-QC02 for proper preparation of analytical standards.

9.1.2. Store all standard solutions in ≤6°C without freezing.

**9.2. Stock Standard Solution**9.2.1. Primary Stock Standards – Purchase a commercially-prepared Potassium Dichromate ( $K_2Cr_2O_7$ ) stock standard, certified solution at 1000 mg/L. This standard is used for initial calibration and continuing calibration.9.2.2. Secondary Stock Standards – Purchase Potassium Dichromate ( $K_2Cr_2O_7$ ) neat standard at 99% purity ACS. Prepare a 1000 mg/L stock standard by dissolving 0.283g to 50mL reagent water and dilute it to 100mL. This standard is used for ICV, and QCS.

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The primary and secondary stock standards can alternatively be a commercially prepared solution or prepared from neat standard provided that these are from two different sources.

9.2.3. Preparation of Intermediate Standard

9.2.3.1. Dilute 1 mL of stock solution to 100 mL reagent water. The expected concentration is 10 mg/L.

9.2.3.2. The primary intermediate standard is prepared from the primary stock standard.

9.2.3.3. The secondary intermediate standard is prepared from the secondary stock standard.

9.2.4. Preparation of Working Standard

9.2.4.1. Dilute 1 mL of intermediate standard to 100 mL reagent water. The expected concentration is 100 µg/L.

9.2.4.2. Prepare the primary working standard from the primary intermediate standard.

9.2.4.3. Prepare the secondary working standard from the secondary intermediate standard.

9.2.5. Preparation of Initial Calibration (ICAL), Quality Control Samples (QCS) and Continuing Calibration Verification (CCV) Standards

9.2.5.1. **Regular Level Concentration**

9.2.5.1.1. Initial calibration standards – Prepare a minimum of three standards and a blank. Using a micropipette, prepare the ICAL standards using the primary working standard solution (100 µg/L) to a final volume of 100mL reagent water (when calibration is used for unpreserved samples) or buffer solution (when calibration is used for preserved samples). Refer to the suggested ICAL standards table below.

ICAL Standard	Primary Working Standard (mL)	Final Analyte Concentration (µg/L)
S0	0	0
S1	0.2	0.2
S2	1.0	1
S3	2.0	2
S4	5.0	5
S5	7.5	7.5
S6	10.0	10

Other concentrations may be prepared to meet project data quality objective as long as ICAL acceptance criteria are met.

***NOTE: Final analyte concentration of the calibration points may vary depending upon the sensitivity of the instrument.***

9.2.5.1.2. Quality Control Samples – Use the secondary working standard (100 µg/L) to spike the Initial Calibration Verification (ICV) at 4 µg/L, and Lab Control Sample (LCS) and Matrix Spike (MS) at 2 µg/L.

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9.2.5.1.3. Continuing Calibration Standard – Use the primary working standard for Continuing Calibration Verification (CCV) at a spike level of 2 µg/L.

9.2.5.2. **Low Level Concentration**

9.2.5.2.1. Initial calibration standards – Prepare a minimum of six standards and a blank. Using a micropipette, prepare the ICAL standards using the primary working standard solution (100 µg/L) to a final volume of 100mL reagent water (when calibration is used for unpreserved samples) or buffer solution (when calibration is used for preserved samples). Refer to the suggested ICAL standards table below.

ICAL Standard	Primary Working Standard (mL)	Final Analyte Concentration (µg/L)
S0	0	0
S1	0.05	0.05
S2	0.1	0.1
S3	0.2	0.2
S4	0.5	0.5
S5	1.0	1
S6	2.0	2
S7	5.0	5
S8	10.0	10

Other concentrations may be prepared to meet project data quality objective as long as ICAL acceptance criteria are met.

***NOTE: Final analyte concentration of the calibration points may vary depending upon the sensitivity of the instrument.***

9.2.5.2.2. Quality Control Samples – Use the secondary working standard (100 µg/L) to spike the ICV at 2 µg/L, and LCS and MS at 1 µg/L.

9.2.5.2.3. Continuing Calibration Standard – Use the primary working standard for CCV at a spike level of 1 µg/L.

## 10.0 **PROCEDURES**

### 10.1. **Sample Preparation For Aqueous Samples**

#### 10.1.1. Unpreserved Aqueous Samples

10.1.1.1. Withdraw the samples from the sample control room and allow the samples to equilibrate to room temperature.

10.1.1.2. Withdraw 40 mL of each sample.

10.1.1.3. Check the sample pH. Record the pH. If the sample pH is not within 9 – 9.5, adjust the pH by drop-by-drop addition of 2.5% NaOH or buffer solution. Record the final pH.

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- 10.1.1.4. Take clean sample vials equal to the number of samples to be analyzed not to exceed 20 field samples. If number of samples are  $\leq 10$  add vials for ICV, ICB, MB, LCS, and MS, If samples  $> 10$  add three more vials for CCV, CCB, and MS.
- 10.1.1.5. Using a 5-mL plastic syringe, withdraw 5 mL of sample and attach the 0.20  $\mu\text{m}$  filter. Discard the first 2 mL. Collect the rest on a properly labeled sample container. For MB and LCS, use reagent water.
- 10.1.1.6. Repeat steps 10.1.1.3 and 10.1.1.5 for all samples to include the MB, LCS, CCB and CCV.
- 10.1.1.7. Spike the LCS and MS designated vials as required either by 9.2.5.1.2 (regular level) or 9.2.5.2.2 (low-level).
- 10.1.1.8. Using a calibrated micropipette, add sufficient amount of sample (i.e., 5 mL for Inst. 59 and D6 vials or 10 mL for G2 vials) into the sample vials and MS vial. Add sufficient amount of reagent water for MB and LCS.
- 10.1.1.9. Seal the vials and shake the QC samples to attain homogeneity on the mixture.

**10.1.2. Preserved Aqueous Samples**

- 10.1.2.1. Withdraw the samples from the sample control room and allow the samples to equilibrate to room temperature.
- 10.1.2.2. Withdraw 40 mL of each sample.
- 10.1.2.3. Check the sample pH. Record the pH. If the sample pH is not within 9 – 9.5, adjust the pH by drop-by-drop addition of 2.5% NaOH or buffer solution. Record the final pH.
- 10.1.2.4. Take clean sample vials equal to the number of samples to be analyzed not to exceed 20 field samples. If number of samples are  $\leq 10$  add vials for ICV, ICB, MB, LCS, and MS, If samples  $> 10$  add three more vials for CCV, CCB, and MS.
- 10.1.2.5. Using a 5-mL plastic syringe, withdraw 5 mL of sample and attach the 0.20  $\mu\text{m}$  filter. Discard the first 2 mL. Collect the rest on a properly labeled sample container. For MB and LCS, use buffer solution.
- 10.1.2.6. Repeat steps 10.1.2.3 and 10.1.2.5 for all samples to include the MB, LCS, CCB and CCV.
- 10.1.2.7. Spike the LCS and MS designated vials as required either by 9.2.5.1.2 (regular level) or 9.2.5.2.2 (low-level).
- 10.1.2.8. Using a calibrated micropipette, add sufficient amount of sample (i.e., 5 mL for Inst. 59 and D6 vials or 10 mL for G2 vials) into the sample vials and MS vial. Add sufficient amount of buffer solution for MB and LCS.
- 10.1.2.9. Seal the vials and shake the QC samples to attain homogeneity on the mixture.

**10.2. Sample Preparations For Soil Sample****10.2.1. Leaching Method**

- 10.2.1.1. Allow sample to equilibrate at room temperature.
- 10.2.1.2. Mix sample thoroughly discarding artifacts, e.g. vegetation, rocks, etc.

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- 10.2.1.3. Tare a 125 mL-polyethylene snap-seal container.
- 10.2.1.4. Weigh 10gm of mixed soil sample to the nearest 0.1gm.
- 10.2.1.5. Using a graduated cylinder, add 100 mL of reagent water to the bottle. Place the bottle in a shaker and mix the solution for 10 minutes.
- 10.2.1.6. Remove containers from the shaker and centrifuge the slurry for 15-30 minutes or allow the particles to settle.
- 10.2.1.7. Treat the liquid phase like an aqueous sample.

10.2.2. Alkaline Digestion

- 10.2.2.1. Refer to EMAX-3060 for procedure.

10.3. **Instrument Parameters**10.3.1. Instruments 59 and D6

	Inst. 59	Inst. D6
Instrument	Dionex AD 25	Dionex ICS-1000
Detector	UV/VIS AD25 Absorbance Detector	UV/VIS AD25 Absorbance Detector
Sample Flow Rate	1.0 mL/min	1.0 mL/min
Isocratic Pump BackPressure	200-2500 psi	200-2500 psi
Sample Loop	250 µL	500 µL
Eluent Pressure	6-9 psi	6-9 psi
Regenerant Pressure	50-70 psi	50-70 psi
Run Time	10 min	15 min
Absorbance Unit Range	0.05 AU	0.05 AU
Diagnostic Test	CPU, LEAK, LAN, wavelength cal, VIS Lamp, UV Lamp, Opitical (Display Pass "P" test)	CPU, LEAK, LAN, wavelength cal, VIS Lamp, UV Lamp, Opitical (Display Pass "P" test)

10.3.2. Instrument G2

Instrument	Metrohm Compact IC Pro881
Detector	Metrohm Professional UV/Vis Detector 887
Eluent Pump Back Pressure	7 -15 MPa
Sample Flow Rate	0.8 mL/min
Regenerant Back Pressure	0.1 – 0.3 MPa
PC Flow Rate	0.22 mL/min
Column Guard Temperature	35 °C

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Sample Loop	2 mL
Run Time	12 min.

**10.4. Calibration****10.4.1. Initial Calibration****10.4.1.1. Instruments 59 and D6**

- 10.4.1.1.1. Set up the IC AD 25 with the proper operation parameter established in Section 10.3.1.
- 10.4.1.1.2. Start the flow of the eluent by pressurizing the eluent reservoir with nitrogen gas to ensure constant delivery to the column. Prime the pump to eliminate air bubbles in the system and equilibrate the column for 30 minutes or until stable baseline is obtained.
- 10.4.1.1.3. Using the Chromeleon data acquisition program in the browser window, open the previous sequence and "Save as" under a new name. Go to the top square in the browser window and rename the method name. Below is an example of a sequence file.

Sequence: IA12  
Operator: EMAXLABSPage 1 of 1  
Printed: 1/15/2009 9:31:59 AM

Title: Temporary sequence for manual data acquisition

Datasource: DG7B3Q91\_local  
Location: DX600/2009  
Timebase: DX600  
#Samples: 10Created: 1/15/2009 9:26:42 AM by EMAXLABS  
Last Update: 1/15/2009 9:27:16 AM by EMAXLABS

No.	Sequence	Sample ID	Name	Type	Inj. Vol.	Program	Method	Status	Inj. Date/Time	Dil. Factor	Comment
1	IA12	001	IB	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
2	IA12	002	S-0	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
3	IA12	003	S-1	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
4	IA12	004	S-2	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
5	IA12	005	S-3	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
6	IA12	006	S-4	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
7	IA12	007	S-5	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
8	IA12	008	S-6	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
9	IA12	009	ICV	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
10	IA12	010	ICB	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	

- 10.4.1.1.4. Set the calibration standards on the Calibration Table as shown in Section 9.2.5.
- 10.4.1.1.5. Place the standards chronologically into the Autosampler rack starting from Instrument Blank (IB).
- 10.4.1.1.6. Go to Browser / Batch and start to initiate analysis of the standards.
- 10.4.1.1.7. After all the standards are analyzed, plot the calibration curve of peak area against concentration and automatically it calculates the correlation coefficient ( $r^2$ ). Go to Report / Calibration (Current Peak) for the calibration curve and the  $r^2$ .

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10.4.1.1.8. Check Appendix 1 for acceptance criteria and corrective action.

10.4.1.2. **Instrument G2**

10.4.1.2.1. Set up G2 with the proper calibration parameter established in Section 10.3.2.

10.4.1.2.2. Turn on the column heater to let the column heat up to 35°C. Select “Prep-Method” on workplace. Prime the system and start the flow of eluent by starting the equilibration process. Equilibrate the system until a stable baseline is obtained.

10.4.1.2.3. Using the Magic Net 2.3 software, open the previous ICAL sequence and “Save As” method under a new name. Update the information in the new sequence. Create new database under database manager. Go to method icon on left side of the screen and open the new method. Click on “Result” icon and update name of the database. Close the method prior to initiating the standard analysis. Below is a sample of the sequence file.

10.4.1.2.4. Set the calibration standards on the Calibration Table as shown in Section 9.2.5.

MagIC Net 2.3 Build 87 - 1  
License ID: 15098587

Client name: G2-PC

User (short name): SAM  
Printed: 2012-11-20 13:33:09 UTC-8

## Determination overview

Determination start	Ident	Sample type	Method name	User (short name)	Info 1
1 2012-08-08 20:12:43 UTC-7	IB	Sample	HCG2H08	LUCY	DH08-01
2 2012-08-08 20:29:14 UTC-7	S0	Sample	HCG2H08	LUCY	DH08-02
3 2012-08-08 20:45:45 UTC-7	S1 0.05PPB WATER	Standard 1	HCG2H08	LUCY	DH08-03
4 2012-08-08 21:02:16 UTC-7	S2 0.10PPB WATER	Standard 2	HCG2H08	LUCY	DH08-04
5 2012-08-08 21:18:46 UTC-7	S3 0.20PPB WATER	Standard 3	HCG2H08	LUCY	DH08-05
6 2012-08-08 21:35:17 UTC-7	S4 0.50PPB WATER	Standard 4	HCG2H08	LUCY	DH08-06
7 2012-08-08 21:51:48 UTC-7	S5 1.0PPB WATER	Standard 5	HCG2H08	LUCY	DH08-07
8 2012-08-08 22:08:17 UTC-7	S6 2.0PPB WATER	Standard 6	HCG2H08	LUCY	DH08-08
9 2012-08-08 22:24:47 UTC-7	S7 5.0PPB WATER	Standard 7	HCG2H08	LUCY	DH08-09
10 2012-08-08 22:41:17 UTC-7	S8 10PPB WATER	Standard 8	HCG2H08	LUCY	DH08-10
11 2012-08-08 23:30:50 UTC-7	ICV 1.0PPB WATER	Sample	HCG2H08	LUCY	DH08-13
12 2012-08-08 23:47:20 UTC-7	ICV 2.0PPB WATER	Sample	HCG2H08	LUCY	DH08-14
13 2012-08-09 00:03:50 UTC-7	ICB WATER	Sample	HCG2H08	LUCY	DH08-15

10.4.1.2.5. Place the standards chronologically into the Autosampler Rack starting from the Instrument Blank (IB).

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- 10.4.1.2.6. Go to workplace / determination series and start to initiate analysis of the standards.
- 10.4.1.2.7. After all the standards are analyzed, select all the standard result from database, and go to reprocess. Integrate the peaks, if necessary, and reprocess the calibration "from standards of reprocessing table". It automatically calculates the correlation coefficient ( $r^2$ ). Select S7 where it has the standard points on standard curve, and reprocess the calibration again "from selected determination".
- 10.4.1.2.8. Check Appendix 1 for acceptance criteria and corrective action.
- 10.4.2. Initial Calibration Verification (ICV)
  - 10.4.2.1. Analyze ICV spiked with QCS after the initial calibration to verify the validity of the initial calibration concentration.
- 10.4.3. Continuing Calibration Verification (CCV)
  - 10.4.3.1. Analyze CCV every 10 samples and at the end of the analytical batch.
- 10.4.4. Retention Time Window (RTW)
  - 10.4.4.1. **Establishing RTW**
    - 10.4.4.1.1. Run RTW standard over a period of 72 hours.
    - 10.4.4.1.2. Calculate the standard deviation (SD) of the absolute retention time obtained for the analyte.
    - 10.4.4.1.3. The width of RTW is defined by  $\pm 3 \times$  SD obtained from 10.4.4.1.2.
  - 10.4.4.2. **Evaluating RTW**
    - 10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained to define the RTW for the current instrument.
    - 10.4.4.2.2. For new instruments, use the established retention time from another instrument having the same instrument parameters (e.g., detector, temperature program and column). If there are no instruments with the same instrument parameter, use 0.03 minutes as the default RTW until historical data is obtained to define the RTW for the current instrument parameter condition.
  - 10.4.4.3. **Application of RTW**
    - 10.4.4.3.1. Establish the center of absolute retention time for the analyte from the daily calibration check at the beginning of the analytical shift then apply the established RTW.
    - 10.4.4.3.2. Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analysis.
  - 10.4.4.4. **Updating RTW**

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10.4.4.4.1. Re-establish the RTW as described in 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g., succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g., replacement of detector or column; temperature program change, etc.)

10.4.4.4.2. If the calculated RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

**10.5. Analysis****10.5.1. Analytical Sequence**

- 10.5.1.1. ICV – initial calibration check
- 10.5.1.2. ICB – initial calibration blank (reagent water)
- 10.5.1.3. MB – Method Blank
- 10.5.1.4. LCS – Lab Control Sample
- 10.5.1.5. MS– matrix spike sample
- 10.5.1.6. Sample Duplicate or MS Duplicate (as may be required by the project)
- 10.5.1.7. Samples – maximum of 10 field samples
- 10.5.1.8. CCV – continuing calibration check
- 10.5.1.9. CCB – continuing calibration blank
- 10.5.1.10. MS
- 10.5.1.11. Sample Duplicate or MS Duplicate
- 10.5.1.12. Samples – maximum of 10 field samples
- 10.5.1.13. CCV
- 10.5.1.14. CCB

**10.5.2. Sample Result Evaluation**

- 10.5.2.1. All samples, including QC samples shall be injected in duplicates. Expected RSD of the duplicate injection is <20%.
- 10.5.2.2. All sample runs, including the Laboratory Reagent Blank (MB), Laboratory Control Sample (LCS), Duplicate Sample (Dup), and Matrix Spike Sample (MS), should be bracketed with calibration checks.
- 10.5.2.3. Check QC results as soon as possible.
- 10.5.2.4. If any analyte concentration exceeds the initial calibration range, perform appropriate dilution to bring the concentration to be within the range and reanalyze the dilution. All re-analyses due to dilution shall be bracketed with continuing calibrations.

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- 10.5.2.5. Check each of the instrument performance checks that it meets the acceptance criteria set forth in Appendix 1.
- 10.5.2.6. Check that the retention time for all positive results fall within the established RTW.
- 10.5.2.7. Check the peaks of all positive results. Refer to Figure 1 for typical peak evaluation.
- The same peak integration technique applied in the initial calibration must be applied during the analysis of field samples.
  - Peaks must be well-resolved and properly integrated.
  - For manual integration, refer to EMAX-DM01 (see section on manual integration).
  - If a peak appears to be cryptic / anomalous, consult the supervisor.
- 10.5.2.8. Check if any of the sample results exceed the calibration range. If so, check that the diluted sample or extract is within the calibration range.
- 10.5.2.9. Rule-out any suspicion of carry-over. Any sample with trace amount of analyte seen in a previous sample that exceeds the calibration range needs to be re-analyzed.
- 10.5.2.10. Check the Lab Reagent Blank (or Method Blank) for absence or presence of contamination.
- 10.5.2.11. Check the Lab Fortified Blank (or Lab Control Sample) for method performance.
- 10.5.2.12. Check the Lab Fortified Matrix (or Matrix Spike) for absence or presence of matrix interference.

**10.6. Calculations**

- 10.6.1. For water samples, if the initial sample taken was  $V_i$  and diluted to  $V_f$  mL, then calculate the concentration using the following equation:

$$C_w = (C_i)(DF) \quad \text{Eq.-10.6.1}$$

where:

- $C_i$  – Concentration of diluted sample, mg/L
- $C_w$  – Concentration of original sample, mg/L
- $DF$  – Dilution Factor;  $DF=V_f/V_i$
- $V_i$  – Initial volume of the diluted sample, mL
- $V_f$  – Final volume of the diluted sample, mL

- 10.6.2. For solid samples, use the following equation:

$$C_s = C_i \times \frac{V_e}{W_s} \times \frac{V_f}{V_i} \times \frac{100}{100 - \%M} \quad \text{Eq.-10.6.2}$$

where:

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$C_s$  – Concentration of the sample based on dry weight, mg/Kg

$C_i$  – Concentration in diluted digestate, mg/L

$V_e$  – Volume of extract, mg/L

$W_s$  – Weight of wet sample, g

$DF$  – Dilution Factor

$\%M$  – Percent Moisture

10.6.3. Calculate for Percent Recovery

$$\% \text{ Recovery} = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.6.3}$$

where:

$C_f$  – Concentration found

$C$  – Concentration of the sample (use 0 for LCS)

$C_s$  – Concentration of spike

10.6.4. Calculate for Precision

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.6.4}$$

where:

$RPD$  – Relative Percent Difference

$C_1$  – Measured concentration of the first sample aliquot

$C_2$  – Measured concentration of the second sample aliquot

10.7. **Data Reduction**

10.7.1. Make a copy of the analysis and sample preparation log.

10.7.2. Print a copy of the sample preparation log for soil (if any).

10.7.3. Highlight the data to be reported.

10.7.4. Print a copy of the raw data and the QC report.

10.7.5. Collate the reportable raw data separating the QC results from the sample results.

10.7.6. Keep all other data generated with the analytical folder marked with "For record only".

10.8. **Report Generation**

10.8.1. Generate Form 1 to contain the sample results using WDBX<sup>1</sup>.exe and MSRBX<sup>1</sup>.exe in series.

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<sup>1</sup> X represents the latest version of the executable file.

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- 10.8.2. Generate Form 3 to contain the summaries of LCS and MS and Sample Duplicate using QCIC .exe and CQ1N.exe.
- 10.8.3. Generate the case narrative using CN X<sup>1</sup>.exe.
- 10.8.4. Assemble the analytical report in the order listed below.
- 10.8.4.1. Case Narrative
- 10.8.4.2. Lab Chronicle
- 10.8.4.3. Sample Results [Form 1, raw data]
- 10.8.4.4. QC Results [MB, LCS, MS each with raw data]
- 10.8.4.5. Calibration [CAL, ICV, IPC, each with raw data]
- 10.8.4.6. Analytical Log
- 10.8.4.7. Sample Preparation Log
- 10.8.4.8. Non-Conformance Report (if any)
- 10.8.5. Submit the analysis package for secondary review.
- 10.9. **Data Review**
- 10.9.1. Check QC Criteria
- 10.9.1.1. Check the analytical log that samples are analyzed in conformance to the QC frequency and all pertinent records are logged.
- 10.9.1.2. Check that the following conform to QC requirement.
- Holding Time
  - Calibrations
  - MB
  - LCS
  - MS / MD (if specified in the PSR)
- 10.9.2. Check Qualitative Identification
- 10.9.2.1. Check the established RTW for the analytical batch that it was done properly.
- 10.9.2.2. Check that positively identified peaks are integrated properly and within the RTW.
- 10.9.2.3. Check that suspicion of carry-over (if any) was ruled out.
- 10.9.3. Check Quantitation
- 10.9.3.1. Since a program generates the sample result forms, check the calculation of one sample result for correctness.
- 10.9.3.2. Check that dilution factors are properly factored in the calculation.
- 10.9.3.3. Check that correct sample amount and/or extract amount is properly factored.
- 10.9.4. Check For Completeness
- 10.9.4.1. Check that all Forms are present with their corresponding raw data.

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10.9.4.2. Check that the case narrative accurately describes what transpired in the analytical process.

**10.10. Preventive Maintenance**

10.10.1. Perform maintenance activity as prescribed below.

<b>Maintenance Activity</b>	<b>Description</b>	<b>Frequency</b>
Verification	Prime system and check pressure	Daily prior to analysis
Detector Maintenance	Inspect flow cell for leaks and verify performance.	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
Line Fittings Check	Check all air lines for crimping or discoloration. Relocate any pinched line. Replace any damaged line.	Daily prior to analysis
LC Pump Maintenance	Replace pump head seal, purge valve seal and piston rod. Perform wear-in procedure and leak test.	Every six months or as necessary
Column Maintenance	Replace column as necessary. Perform pressure test.	As necessary
Valve Maintenance	Replace pump seal. Inspect valve fittings and capillaries for leaks.	As necessary
System Cleaning	Remove dust from fans and vent covers.	Every six months or as necessary
Sampler Maintenance	Inspect flow rate for proper delivery.	Once a year or as necessary
Inspection	Perform general inspection of the complete system	Once a year

10.10.2. Record all maintenance activities in the Instrument Maintenance Log. Refer to Form 7199FM – Instrument Maintenance Log.

**11.0 QUALITY CONTROL**

11.1. Initial Demonstration of Performance (IDP) shall be accomplished prior to implementation of this procedure and for each analyst that will perform the method. IDP shall constitute the successful completion of the following:

- Initial Calibration (ICAL) and Linear Calibration Range (LCR)
- Analysis of a minimum of four (4) Lab Control Samples (LCS)
- Method Detection Limit (MDL)

11.2. Assessing Laboratory Performance shall be demonstrated in every analytical batch and shall consist of the following QCs verified to be compliant with method and project-specific requirements.

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

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- Method Blank (MB)
  - Lab Control Sample (LCS)
  - Instrument Performance Check (IPC)
  - Each sample is injected twice and expected RSD <20%.
- 11.3. Assessing Analyte Recovery Data Quality on a given matrix shall be demonstrated for each group of sample with similar matrix and shall be compliant with method and project-specific requirements.
- Lab Sample Matrix Recovery (MS)
  - MS spike level must be high enough to be detected above the original sample and should not be less than 4 times the MDL.
  - If the concentration of the fortification is less than 10% of the background concentrations measured in the unfortified sample, the matrix recovery should not be calculated.
  - If the recovery for MS falls outside the recovery range and the LCS is within control, the recovery problem for MS is suspected to be matrix-related not system-related.
- 11.4. LOQ and LOD verification shall be performed every 3 months.
- 11.5. Refer to Appendix 1 for all related Quality Control parameters, frequency, acceptance criteria and corrective action.

**12.0 CORRECTIVE ACTION**

12.1. Implement corrective action as described in Appendix 1.

**12.2. Sample Preparation QC**

12.2.1. For insufficient amount of sample, inform the PM immediately for further action.

12.2.2. When MB is non-compliant,

- Investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.
- If the reagent water shows contamination in the instrument blank or MB, consider changing the filters of the reagent water source.
- If the analyte found in the MB is not detected in any of the field samples, consult the Supervisor and the PM if the result can be reported. Otherwise, reanalyze the MB with the associated samples.

**12.3. Sample Analysis QC**

12.3.1. When Instrument Performance Check (IPC) is non-compliant and all measures (e.g., flushing the column and/or changing the column, etc.) had been undertaken to correct the problem, consult the Supervisor for further advice prior to performing a new ICAL.

12.3.2. When flushing does not get rid of carry-over, consider changing the column.

**12.4. Method QC**

## STANDARD OPERATING PROCEDURES

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12.4.1. When LOD and LOQ verification is non-compliant, consider instrument maintenance and/or reestablish LOD and LOQ. Refer to EMAX-QA04.

12.4.2. When retention time significantly shifts, check for bubbles or leaks.

12.5. **Non-Conformance Report (NCR)**

12.5.1. Refer to EMAX-QA08 for details.

12.5.2. NCR is required when the following circumstances occurred:

- Anomaly other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

**13.0 POLLUTION PREVENTION**

13.1. Quantity of chemicals purchased should be based on expected usage during its shelf life to minimize disposal of unused material.

13.2. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

13.3. Observe all necessary precautions to avoid spillage of reagents that may go to the wastewater drains.

**14.0 WASTE MANAGEMENT**

14.1. Collect all waste generated and properly turn them over to the waste disposal unit.

**15.0 SUPPLEMENTARY NOTES**

15.1. **Definition of Terms**

15.1.1. Analytical batch – is composed of a complete analysis for a batch of no more than 20 field samples. Every 10 field samples or a fraction thereof shall be bracketed with continuing calibration and one LFM is analyzed. For every analytical batch at least one LRB and one LFB is analyzed.

15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.3. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.4. Instrument Performance Check (IPC) – is a mid-range check standard containing the target analytes that is analyzed to verify the instrument calibration at the given criteria.

15.1.5. Laboratory Fortified Blank (LFB) – Also known as Laboratory Control Sample (LCS), is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LFB is analyzed to monitor the accuracy of the analytical system.

15.1.6. Laboratory Fortified Sample Matrix (LFM) – Also known as Matrix Spike (MS), is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample

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preparation and/or analytical process. LFM is analyzed to monitor matrix effect on a method's recovery efficiency.

- 15.1.7. Laboratory Reagent Blank (LRB) – Also known as Method Blank (MB), is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.8. Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.
- 15.1.9. Matrix – is a component or form of a sample.
- 15.1.10. Quality Control Sample (QCS) – A solution obtained from a secondary source different from the source of calibration standard with known concentration of method analytes that is use to fortify an aliquot of ICV, LCS or MS.
- 15.1.11. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.
- 15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

**15.2. Application of QC Procedures**

- 15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing this method. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

**15.3. Department of Defense (DoD) Projects**

- 15.3.1. Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

**15.4. Department of Energy (DoE) Projects**

- 15.4.1. Samples from DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, is applied

**16.0 REFERENCES**

- 16.1. EPA SW846 Method 7199, Method 7199, December 1996.
- 16.2. EPA 218.7, Version 1.0, November 2011.
- 16.3. EMAX Quality Systems Manual, as updated.

**17.0 APPENDICES**

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

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**17.1. Figures**

- 17.1.1. Figure 1 Typical Peak Evaluation
- 17.1.2. Figure 2 Typical Chromatogram
- 17.1.3. Figure 3 Typical ICAL Summary
- 17.1.4. Figure 4 Typical Sample Result
- 17.1.5. Figure 5 Typical LCS/LCSD Summary
- 17.1.6. Figure 6 Typical MS/MSD Summary
- 17.1.7. Figure 7 Typical Sample Duplicate Result Summary
- 17.1.8. Figure 8 Typical Case Narrative

**17.2. Appendices**

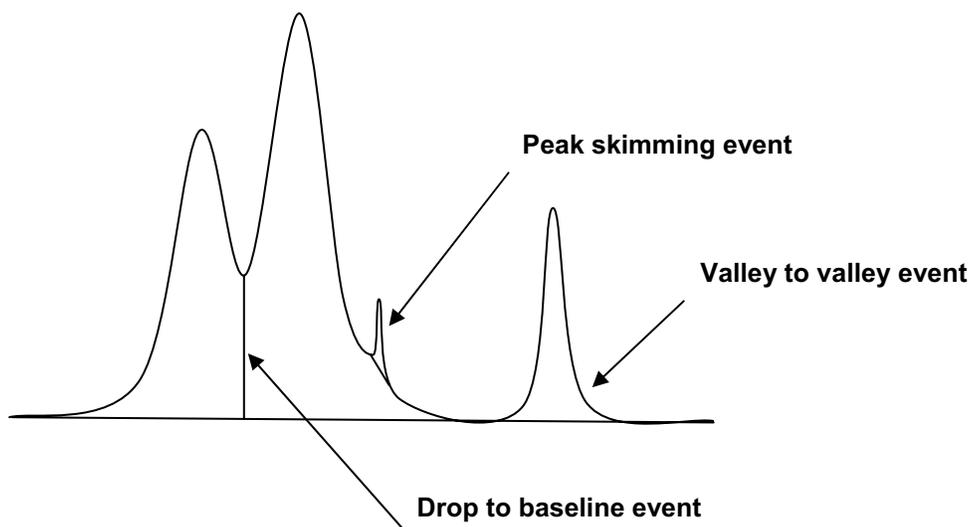
- 17.2.1. Appendix 1A Summary of Quality Control Procedures – Standard
- 17.2.2. Appendix 1B Summary of Quality Control – Low Level
- 17.2.3. Appendix 2 Demonstration of Capability

**17.3. Forms**

- 17.3.1. 7199FSa Sample Preparation Log (Soil Samples)
- 17.3.2. 7199FSb Sample Preparation Log (Water/Preserved Samples)
- 17.3.3. 7199FA Analysis Run Log
- 17.3.4. 7199FM Instrument Maintenance Log

**Figure 1:** **TYPICAL PEAK EVALUATION**

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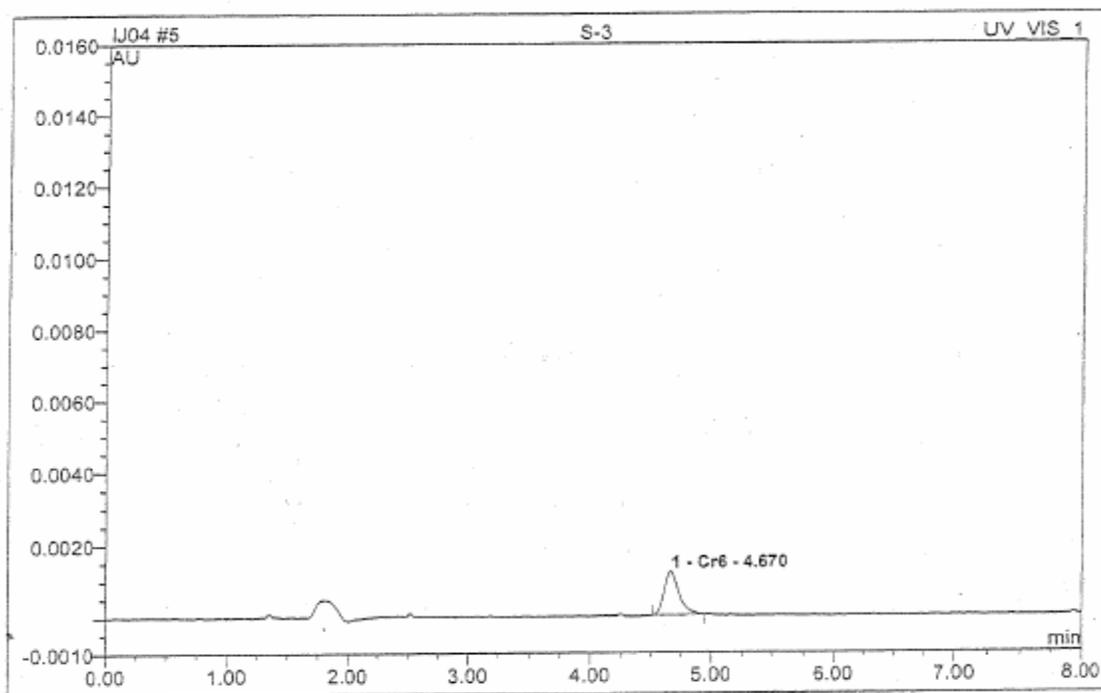


**Figure 2: TYPICAL CHROMATOGRAM**

Operator:EMAXLABS Timebase:DX600 Sequence:IJ04

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<b>IJ04 005 S-3</b>			
Sample Name:	S-3	Injection Volume:	250.0
Vial Number:	0	Channel:	UV_VIS_1
Sample Type:	standard	Wavelength:	n.a.
Control Program:	Cr6 Program	Bandwidth:	n.a.
Quantif. Method:	IC59J04	Dilution Factor:	1
Recording Time:	10/4/2012 12:27	Sample Weight:	1.0000
Run Time (min):	8.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height AU	Area AU*min	Rel.Area %	Amount	Type
1	4.67	Cr6	0.0012030	0.0001733	100.00	1.966	BMB
Total:			0.001	0.000	100.00	1.966	

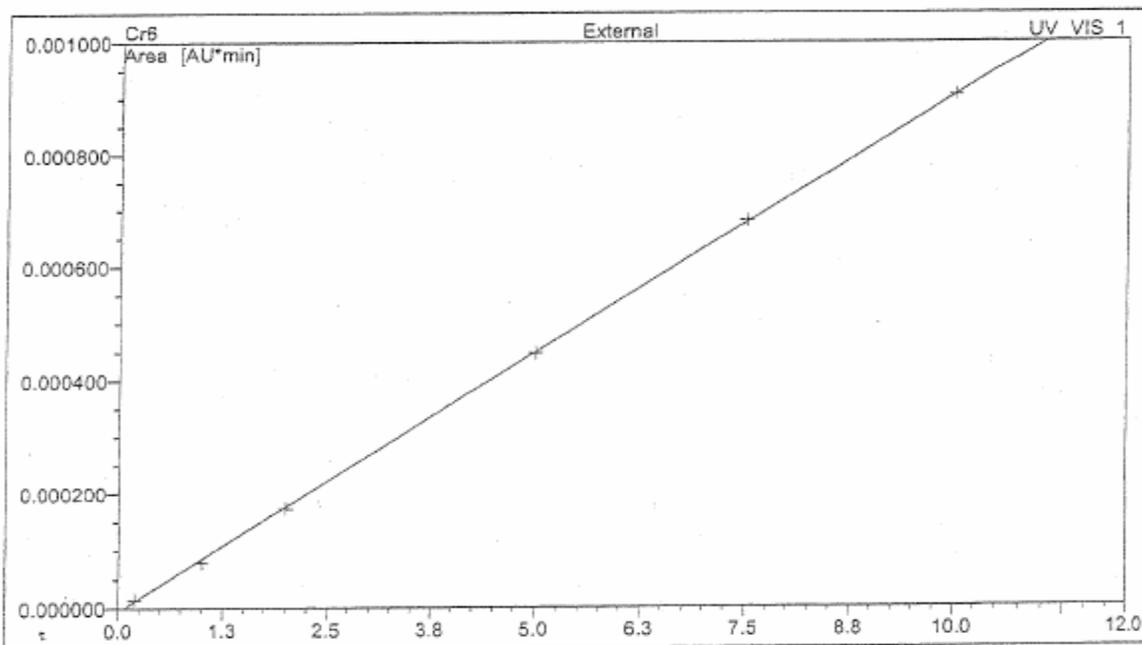
*AS*  
*09/11/2*

**Figure 3: TYPICAL ICAL SUMMARY**

Operator:EMAXLABS Timebase:DX600 Sequence:IJ04

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<b>8 S-6</b>			
Sample Name:	S-6	Injection Volume:	250.0
Vial Number:	0	Channel:	UV_VIS_1
Sample Type:	standard	Wavelength:	n.a.
Control Program:	Cr6 Program	Bandwidth:	n.a.
Quantif. Method:	IC59J04	Dilution Factor:	1.0000
Recording Time:	10/4/2012 12:59	Sample Weight:	1.0000
Run Time (min):	8.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Cal.Type	Points	R-Square	Offset	Slope	Curve
1	4.66	Cr6	0LOff	6	0.9999	-0.0000062	0.0000913	0.0000
Average:					0.9999	0.0000	0.0001	0.0000

*At*  
*10/9/12*

**Figure 4: TYPICAL SAMPLE RESULT**

METHOD 7199  
 HEXAVALENT CHROMIUM

```

=====
Client       : XYZ INC.
Project      : CLEAN WATER
Batch No.   : 12J056
Matrix      : WATER
Instrument ID : I59
=====
  
```

SAMPLE ID	EMAX SAMPLE ID	RESULTS (ug/L)	DLF	MOIST	LOQ (ug/L)	LOD (ug/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK1W	HCJ004WB	ND	1	NA	0.200	0.100	10/09/1212:45	NA	IJ09003	IJ09001	HCJ004W	NA	NA
LCS1W	HCJ004WL	2.08	1	NA	0.200	0.100	10/09/1213:06	NA	IJ09005	IJ09001	HCJ004W	NA	NA
LCD1W	HCJ004WC	2.03	1	NA	0.200	0.100	10/09/1213:27	NA	IJ09007	IJ09001	HCJ004W	NA	NA
129178-25-0038	J056-01	1.31	1	NA	0.200	0.100	10/09/1217:49	NA	IJ09011	IJ09009	HCJ004W	10/09/1208:53	10/09/12
129178-25-0040	J056-03	0.727	1	NA	0.200	0.100	10/09/1218:10	NA	IJ09013	IJ09009	HCJ004W	10/09/1209:29	10/09/12
129178-25-0040DUP	J056-03D	0.710	1	NA	0.200	0.100	10/09/1218:31	NA	IJ09015	IJ09009	HCJ004W	10/09/1209:29	10/09/12
129178-25-0040MS	J056-03M	2.75	1	NA	0.200	0.100	10/09/1219:13	NA	IJ09019	IJ09016	HCJ004W	10/09/1209:29	10/09/12
129178-25-0040MSD	J056-03S	2.70	1	NA	0.200	0.100	10/09/1219:33	NA	IJ09021	IJ09016	HCJ004W	10/09/1209:29	10/09/12

**Figure 5: TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC  
 PROJECT: CLEAN WATER  
 BATCH NO.: 12J056  
 METHOD: METHOD 7199

=====

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	HCJ004WB	HCJ004WL	HCJ004WC		
LAB FILE ID:	IJ09003	IJ09005	IJ09007		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	NA
DATE ANALYZED:	10/09/1212:45	10/09/1213:06	10/09/1213:27	DATE RECEIVED:	NA
PREP. BATCH:	HCJ004W	HCJ004W	HCJ004W		
CALIB. REF:	IJ09001	IJ09001	IJ09001		

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Hexavalent Chromium	ND	2	2.08	104	2	2.03	102	2	80-120	20

Figure 6:

TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC  
 PROJECT: CLEAN WATER  
 BATCH NO.: 12J056  
 METHOD: METHOD 7199

MATRIX: WATER % MOISTURE: NA  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: 129178-25-0040  
 LAB SAMP ID: J056-03 J056-03M J056-03S  
 LAB FILE ID: IJ09013 IJ09019 IJ09021  
 DATE EXTRACTED: NA NA NA DATE COLLECTED: 10/09/12 09:29  
 DATE ANALYZED: 10/09/1218:10 10/09/1219:13 10/09/1219:33 DATE RECEIVED: 10/09/12  
 PREP. BATCH: HCJ004W HCJ004W HCJ004W  
 CALIB. REF: IJ09009 IJ09016 IJ09016

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	SPIKE AMT (ug/L)	MS RSLT (ug/L)	MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)	MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Hexavalent Chromium	0.727	2	2.75	101	2	2.70	99	2	75-125	20

**Figure 7: TYPICAL SAMPLE DUPLICATE RESULT SUMMARY**

EMAX QUALITY CONTROL DATA  
 DUPLICATE SAMPLE ANALYSIS

CLIENT: XYZ, INC  
 PROJECT: CLEAN WATER  
 BATCH NO.: 12J056  
 METHOD: METHOD 7199

-----  
 MATRIX: WATER % MOISTURE: NA  
 DILUTION FACTOR: 1  
 SAMPLE ID: 129178-25-0040  
 EMAX SAMP ID: J056-03 J056-03D  
 LAB FILE ID: IJ09013 IJ09015  
 DATE EXTRACTED: NA NA DATE COLLECTED: 10/09/12 09:29  
 DATE ANALYZED: 10/09/1218:10 10/09/1218:31 DATE RECEIVED: 10/09/12  
 PREP. BATCH: HCJ004w HCJ004w  
 CALIB. REF: IJ09009 IJ09009

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	DUPL RSLT (ug/L)	RPD RSLT %	QC LIMIT ( % )
Hexavalent Chromium	0.727	0.710	2	20

**Figure 8: TYPICAL CASE NARRATIVE**

---

CASE NARRATIVE

Client : XYZ, INC  
Project : CLEAN WATER  
SDG : 12J056

METHOD 7199  
HEXAVALENT CHROMIUM

A total of two (2) water samples were received on 10/09/12 for Hexavalent Chromium by IC analysis, Method 7199 in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source. Continuing calibration verifications were carried out at the frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for HCJ004WL/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at the frequency prescribed by the project. Percent recoveries for J056-03M/S were within project QC limits. Sample duplicate was also analyzed with the samples. RPD was within project limit.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter.

**Appendix 1A: SUMMARY OF QUALITY CONTROL PROCEDURES – STANDARD**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Minimum 3-pt + blank calibration for all analytes (ICAL)	When IPC fails to meet the acceptance criteria.	Correlation coefficient $\geq 0.999$ for linear regression	Locate and correct the source of problem, then perform ICAL.		
Quality Control Sample (QCS) secondary source	After ICAL and every beginning of analytical batch	All analytes within $\pm 10\%$ of expected value	Repeat the analysis, if problem persist locate and correct the source of problem, then perform ICAL.		
Continuing Calibration CCV/CCB	Every 10 analyses	CCV: All analytes within $\pm 10\%$ of expected value CCB: $\leq \frac{1}{2}$ LOQ	Repeat the analysis, if problem persist locate and correct the source of problem, then perform ICAL.		
Replicate Injection	All samples including standards, QC samples and field samples	RPD: $<20\%$	Repeat the analysis. If problem persist discuss in the case narrative.		
Method Blank (MB)	One per preparation batch	No analyte detected $> \frac{1}{2}$ LOQ	Re-prep and reanalyze MB and all samples processed with MB.		
Lab Control Sample (LCS)	One per preparation batch	%Recovery: 80 – 120%	Re-prep and reanalyze the LCS and all associated samples.		
Matrix QC Sample (MS)	One per 10 field samples per matrix	MS %Recovery : 75 –125%	If LCS passed, no action.		
Sample Duplicate or Matrix Sample Duplicate (MSD)	One per 10 field samples per matrix	RPD: $\leq 20\%$	If LCS passed, no action.		
Comments: Refer to PSR for Flagging Criteria.			Reviewed By:		
			Date:		

**Appendix 1B: SUMMARY OF QUALITY CONTROL PROCEDURES – LOW LEVEL**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Minimum 6-pt + blank calibration for all analytes (ICAL)	When IPC fails to meet the acceptance criteria.	Correlation coefficient $\geq 0.999$ for linear regression	Locate and correct the source of problem, then perform ICAL.		
Quality Control Sample (QCS) secondary source	After ICAL and every beginning of analytical batch	All analytes within $\pm 10\%$ of expected value	Repeat the analysis, if problem persist locate and correct the source of problem, then perform ICAL.		
Continuing Calibration CCV/CCB	Every 10 analyses	CCV: All analytes within $\pm 10\%$ of expected value CCB: $\leq \frac{1}{2}$ LOQ	Repeat the analysis, if problem persist locate and correct the source of problem, then perform ICAL.		
Replicate Injection	All samples including standards, QC samples and field samples	RPD: $<20\%$	Repeat the analysis. If problem persist discuss in the case narrative.		
Method Blank (MB)	One per preparation batch	No analyte detected $> \frac{1}{2}$ LOQ	Re-prep and reanalyze MB and all samples processed with MB.		
Lab Control Sample (LCS)	One per preparation batch	%Recovery: 80 – 120%	Re-prep and reanalyze the LCS and all associated samples.		
Matrix QC Sample (MS)	One per 10 field samples per matrix	MS %Recovery : 75 –125%	If LCS passed, no action.		
Sample Duplicate or Matrix Sample Duplicate (MSD)	One per 10 field samples per matrix	RPD: $\leq 20\%$	If LCS passed, no action.		
Comments: Refer to PSR for Flagging Criteria.			Reviewed By:		
			Date:		

**APPENDIX 2: DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
 METHOD EPA 7199**

**MATRIX: WATER**

Unit: µg/L  
 Instrument ID: IC59

Date Analyzed: 06/22/12  
 Analyzed By: Viet Le

PARAMETER	HCF025WL IF23015	HCF025WC IF23017	HCF027WL IF23019	HCI010WC IF23021	True Value	Ave. Conc.	Ave. % Rec.	SD	RSD	QC Criteria	COMMENTS
Hexavalent Chromium	1.896	1.889	1.897	1.888	2	1.91	96	0.005	0.24	80 - 120	Passed

**MATRIX: SOIL**

Unit: mg/Kg  
 Instrument ID: IC59

Date Analyzed: 07/19/12  
 Analyzed By: Viet Le

**EXTRACTION BY EPA 3060A**

PARAMETER	CSG006SL IG20005	CSG006SC IG20007	CSG007SL IG20009	CSG007SC IG20011	True Value	Ave. Conc.	Ave. % Rec.	SD	RSD	QC Criteria	COMMENTS
Hexavalent Chromium	8.550	8.520	8.500	8.535	10	8.82	88	0.021	0.24	80 - 120	Passed



7199FSb:

**SAMPLE PREPARATION LOG (WATER/PRESERVED SAMPLES)**

Page 1



**pH ADJUSTMENT/SAMPLE PRESERVATION LOG  
FOR HEXAVALENT CHROMIUM**

SOP  EMAX-218.6 Rev. 5  EMAX-7199 Rev. 2

Book #: PHC-006

Matrix:		Start Date:		Time:		End Date:		Time:	
Sample Prep ID	Lab Sample ID	Initial pH	Final pH	Notes		Standards	ID	pH	
01						Buffer 7			
02						Buffer 10			
03						Check pH Buffer (8)			
04						pH meter			
05									
06						Reagent	Standard ID #		
07						NaOH			
08						(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> /NH <sub>4</sub> OH			
09						Reagent Water			
10						Legend:			
11						Color	Texture	Clarity	Artifacts
12						Bu = Blue Bl = Black	Cs = Coarse	Cr = Clear	Rk = Rocks
13						Bn = Brown Gn = Green	Md = Medium	Cy = Cloudy	Sl = Shale
14						Og = Orange Rd = Red	Fn = Fine	Td = Turbid	Vg = Vegetation
15						Yw = Yellow			
16						<input type="checkbox"/> Samples were received with preservation			
17						Comments: _____			
18						_____			
19						_____			
20						_____			
21						_____			
22						_____			
23						Prepared By: _____			
24						Standard Added By: _____			
25						Checked By: _____			

7199FA:

ANALYSIS RUN LOG



ANALYSIS RUN LOG  
*for*  
 HEXAVALENT CHROMIUM IC

Page 1

**Note:** For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

**Comments:**

**Post Column Solution:**

1,5 Diphenylcarbohydrozide : 0.5g

MeOH : 100 mL

H<sub>2</sub>SO<sub>4</sub> : 28 mL

**Eluent:**

Book #: AG2-003

Instrument No.: G2

Pipette ID: SW8A-02-19

SW8A-02-20

SW8A-02-21

Balance ID: 40706360

Analytical Sequence:

Method File:

Analytical Batch:

SOP #	Rev. #
<input type="checkbox"/> EMAX-218.6	5
<input type="checkbox"/> EMAX-7199	2
<input type="checkbox"/> EMAX-	

STANDARDS ID	
ICAL	
ICV	
CCV	
LCS	
MS	

ELECTRONIC DATA ARCHIVAL	
Location	Date
<input type="checkbox"/> External Hard Drive	
<input type="checkbox"/>	

Analyzed By: \_\_\_\_\_

Date: \_\_\_\_\_



SOP REVIEW FORM

EMAX-7470

SOP No.

Rev. 7

Revision Number

MERCURY IN LIQUID WASTE

Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read the following MSDS:
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

Mary Jane Mendoza *fm*

Date:

7/8/14

## STANDARD OPERATING PROCEDURES

**MERCURY IN LIQUID WASTE**

SOP No.: EMAX-7470 Revision No. 7 Effective Date: 12-Dec-12

Prepared By: Mary Jane Mendoza *gjm* Date: 12.11.12

Approved By: Kenette Pimentel *[Signature]* Date: 12.11.12  
QA Manager

Approved By: Caspar Pang *[Signature]* Date: 12-11-12  
Laboratory Director

Control Number: 7470-07-

**1.0 SCOPE AND APPLICATION**

- 1.1. This procedure applies to the measurement of Mercury in aqueous wastes, leachates, and wastewater samples by Cold Vapor Absorption Technique.
- 1.2. This SOP is an adaptation of SW846 Methods 7470A.

**2.0 SUMMARY OF METHOD**

- 2.1. A representative amount of sample is digested in nitric and sulfuric acids, followed by oxidation with potassium permanganate and potassium persulfate.
- 2.2. Organic mercurial are broken down and converted into mercuric ions in order to respond to the cold vapor atomic absorption technique. Persulfate oxidation step, followed by addition of permanganate ensures that organo-mercury compounds are oxidized.
- 2.3. Absorption of radiation by mercury vapor at 253.7 nm is then measured in the digested samples.
- 2.4. **Interferences**
  - 2.4.1. Sulfides, as sodium sulfide, Copper and Chloride at high concentrations are known to interfere with the recovery of mercury. Samples containing such interference may require additional permanganate (about 12.5 ml).
  - 2.4.2. Care must be taken to ensure that free chlorine is absent before the mercury is swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent.
  - 2.4.3. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

**3.0 DETECTION LIMITS**

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) and Limit of Quantitation (LOQ)**
  - 3.1.1. Refer to EMAX-QA04 for generation, validation and verification of DL, LOD and LOQ.
- 3.2. **Established DL, LOD & LOQ**

PARAMETER	DL	LOD	LOQ	Unit
Water	0.054	0.1	0.50	µg/L

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**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.6). All samples analyzed above this range are considered “over range” and requires dilution to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range are considered as “under range” and requires lower dilution factor to properly quantitate.

**5.0 SAMPLE HOLDING TIME AND PRESERVATION****5.1. Sample Collection**

- 5.1.1. Samples are expected to be contained in HDPE pre-cleaned containers and preserved with HNO<sub>3</sub> to pH < 2 and cooled to ≤ 6°C without freezing.

**5.2. Holding Time**

- 5.2.1. Samples must be analyzed within 28 days from date of collection.

**5.3. Preservation**

- 5.3.1. Store samples and extracts at ≤ 6°C without freezing.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA04 Detection Limit (DL)
- 6.3. EMAX-QA05 Training
- 6.4. EMAX-QA08 Corrective Action
- 6.5. EMAX-QC01 Quality Control of Chemicals
- 6.6. EMAX-QC02 Analytical Standard Preparation
- 6.7. EMAX-QC06 Calibration of Micropipettes
- 6.8. EMAX-QC07 Glassware Cleaning
- 6.9. EMAX-SM03 Waste Disposal
- 6.10. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all MSDS of chemicals listed in this SOP.

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- 7.2. Treat reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure. Perform preparation and analysis of mercury in a fume hood equipped with an exhaust fan or blower.
- 7.3. If for any reason, sample and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist inform your supervisor immediately so that proper action can be taken.
- 7.4. Do not look directly at the Mercury Lamp while lit. The radiation may cause damage to your eyes.
- 7.5. Perform all reagent additions under a fume hood.
- 7.6. Mercury Analyzers are to be used by trained personnel only.

**8.0 INSTRUMENTS, CHEMICALS AND REAGENTS****8.1. Instruments and Supplies**

## 8.1.1. Mercury Analyzers

8.1.1.1. Leeman PS-200 Automated Mercury Analyzer with Autosampler, Computer, Printer and PS 200 Software

8.1.1.2. Leeman Hydra AA Automated Analyzer with Autosampler, Computer, Printer and PS200 Software

8.1.2. 100 ml Digestion vessel

8.1.3. Digestate containers

8.1.4. Digestion block or equivalent

8.1.5. Magnetic stirrer

8.1.6. Micropipettes and tips

8.1.7. Thermometer

**8.2. Chemicals and Reagents**

8.2.1. Where available, purchase reagent grade chemicals and reagents

8.2.2. Sulfuric Acid, concentrated

8.2.3. Nitric Acid, concentrated

8.2.4. Hydroxylamine hydrochloride: Dissolve 120 g of hydroxylamine hydrochloride in reagent water and dilute to 1 L.

8.2.5. Potassium Permanganate, 5% solution: Dissolve 50 g of potassium permanganate in 1 L reagent water.

8.2.6. Potassium Persulfate, 5% solution: Dissolve 50 g of potassium persulfate in 1 L reagent water.

8.2.7. Stannous Chloride – 10% solution: Dissolve 200 g of SnCl<sub>2</sub> in reagent water, add 200 ml HCl and volume to 2 L.

8.2.8. Reagent Water – Mercury-free water

## STANDARD OPERATING PROCEDURES

**MERCURY IN LIQUID WASTE**SOP No.: EMAX-7470 Revision No. 7 Effective Date: 12-Dec-12**9.0 STANDARDS**

- 9.1. Refer to EMAX-QC02 for proper analytical standard preparations.
- 9.2. Other concentration levels may be prepared to meet the data quality objective of a project.
- 9.3. **Stock Standard**
- 9.3.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.
- 9.3.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10°C to -20°C.
- 9.3.3. Prepare calibration standards from the primary standard.
- 9.3.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

Stock Std	Name	Source	CAT #	CONC	NOTES
Primary/CCV	Mercury	Leeman	602-00064	100 mg/L	Or equivalent
ICV /LCS/MS	Mercury	ERA	027	1000 mg/L	Or equivalent

- 9.4. **Intermediate Standard Solution**
- 9.4.1. From 100 µg/L stock solution take a 2 ml aliquot and dilute to 200 ml using reagent water. The solution shall have a final concentration of 1.0 mg/L.
- 9.4.2. Prepare secondary dilution from 1000 mg/L stock solution take a 1 ml aliquot and dilute to 100 ml using reagent water. This solution shall have a final concentration of 10 mg/L.
- 9.5. **Working Standard**
- 9.5.1. From the secondary dilution of intermediate standard prepare the working standard solution to have a final concentration of 50 µg/L.

**9.6. Initial Calibration Standards**

- 9.6.1. From the working solution prepare the following *Leeman* standards in 100 ml volumetric flasks.

Level	Aliquot (ml)	Final Volume (ml)	Concentration (µg/L)
S0	0	50	0
S1	0.2	50	0.2
S2	1.0	50	1.0
S3	2.0	50	2.0
S4	5.0	50	5.0
S5	10.0	50	10.0
CCV	5.0	50	5.0

**9.7. ICV/CCV/LCS/MS**

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**MERCURY IN LIQUID WASTE**SOP No.: EMAX-7470 Revision No. 7 Effective Date: 12-Dec-129.7.1. From the working standard, prepare ICV/CCV/LCS/MS solutions using **ERA** Standards.

Name	Aliquot (ml)	Final Volume (ml)	Concentration (µg/L)
ICV	2.0	50	2.0
LCS/MS	5.0	50	5.0

**10.0 PROCEDURES****10.1. Sample Preparation**

- 10.1.1. Transfer 50 ml of sample into 100 ml digestion vessel. Use reagent water for method blank, LCS, and calibration standards. For STLC and TCLP extracts, use 5 ml sample volume diluted with reagent water to 50 ml. (The reduction of the volume is due to waste minimization.)
- 10.1.2. Add spike standards to LCS/MS. Add appropriate standards for calibration standards. Subsequently perform the following steps for each of the prepared analytical samples.
- 10.1.3. Add 2.5 ml of concentrated H<sub>2</sub>SO<sub>4</sub> and 1.25 ml concentrated HNO<sub>3</sub> with mixing after each addition.
- 10.1.4. Add 7.5 ml of 5% KMnO<sub>4</sub> solution to each vessel.
- 10.1.5. Swirl each vessel to mix and let it stand by for 15 min. Check each vessel if purple color persist. If not, add permanganate solution at 2.5 ml increments swirling the Digestion vessel at every addition, until purple color persists.
- Add the maximum amount of permanganate solution added to a sample, to the method blank, LCS, calibration standards and calibration verification standards.*
- 10.1.6. Add 4 ml of 5% potassium persulfate. Heat for 2 hours in hot block maintained at 95°C.
- 10.1.7. Allow the samples to cool.
- 10.1.8. Add 3 ml hydroxylamine hydrochloride solution and dilute to 80 ml using reagent water. Proceed to 10.2.
- 10.1.9. Properly fill up the sample preparation log.

**10.2. Instrument Parameters****10.2.1. PROTOCOL****10.2.1.1. Set Values**

Instrument ID:	PS200	HYDRA AA
Number of Integration	1	
Uptake time	20 sec.	18 sec.
Weight	N	N
Dilution	N	N
On/Off, times, gains		
On	Y	Y

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Time	10	10
Instrument ID:	PS200	HYDRA AA
Gas	0.35 LPM	0.15 LPM
Pump Rate	5 ml/min	7 ml/min
AUTOSAMPLER – Setup		
Station 1 (rack1)	From cup 1 to cup 44	From cup 1 to cup 44
Station 2 (rack 2)	From cup 1 to cup 44	From cup 1 to cup 44
Rinse time	60 sec.	60 sec.

CALIBRATION	Concentration, µg/L	Concentration, µg/L
S0, S1, S2, S3, S4, S5	0.0,0.2, 1.0, 2.0, 5.0, 10.0	0.0, 0.20, 1.0, 2.0, 5.0, 10.0

## 10.2.2. DATA OUTPUT – Specify Report

Data Output	Real Time	Post Run
Samples	Y	Y
Standards	Y	Y
Updates	Y	Y
Peaks	N	N
IEC Stds.	N	N
Check Stds.	Y	Y
Dups and % Diff.	Y	Y
Wavelength	N	N
Rel. Absorbances	N	N
% RSD	Y	Y
Scans to PRN		N
Detail		Y
Summary		N
Post Run Copies		1
Post Run Report Order [ 1-sorted; 2- sequential]	2	

## 10.3. Calibration

10.3.1. Instrument Set-up

## STANDARD OPERATING PROCEDURES

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10.3.1.1. Set up PS200 or Hydra AA to proper operating parameters. Refer to Section 10.2.1.

*New pump tubing, must be ran with rinse for 45 minutes to break in the tubing.*

10.3.1.2. Turn on the lamp and allow to warm up for at least 5 minutes.

10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.1.4. Check that the reductant solution, 10% SnCl<sub>2</sub>, is sufficient. If not, prepare solution as described in Section 8.2.7.

10.3.2. Initial Calibration (ICAL)

10.3.2.1. Prepare initial calibration solution as described in Section 9.6.1.

10.3.2.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.

10.3.2.3. Analyze them as described in Section 10.4.

10.3.2.4. Refer to Section 10.5 for calculation.

10.3.2.5. Initiate initial calibration as described in the instrument operations manual and acquire the calibration data for review after calibration is completed.

10.3.2.6. Refer to Appendix 1 for acceptance criteria.

10.3.2.7. Verify the initial calibration by a secondary source standard.

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Prepare ICV as described in Section 9.7.1.

10.3.3.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.

10.3.3.3. Analyze the ICV sample to verify the concentration of the ICAL.

10.3.3.4. Refer to Appendix 1 for acceptance criteria.

10.3.4. Continuing Calibration Verification (CCV)

10.3.4.1. Prepare CCV as described in Section 9.6.1.

10.3.4.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.

10.3.4.3. Analyze CCV sample to verify the validity of ICAL.

10.3.4.4. Refer to Appendix 1 for acceptance criteria.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. From the main menu, select Autosampler and the set-up.

10.4.1.1.1. Type the rack number to be ran (1 or 2) and type the rack name and press ENTER. SET UP screen for that rack will appear and a BEGIN CUP prompt will be displayed at the bottom of the screen.

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- 10.4.1.1.2. Enter the number (CUP POSITION) of the first cup to be sampled and press ENTER. An END CUP prompt will now be displayed at the bottom of the screen.
- 10.4.1.1.3. Enter the number of the last cup to be sampled and press ENTER. If second rack is to be used, repeat Steps 10.4.1.1.1. to 10.4.1.1.3.
- 10.4.1.2. Typical Analytical Sequence
  - 10.4.1.2.1. ICV
  - 10.4.1.2.2. ICB
  - 10.4.1.2.3. CCV1
  - 10.4.1.2.4. CCB1
  - 10.4.1.2.5. Method Blank
  - 10.4.1.2.6. LCS
  - 10.4.1.2.7. LCSD
  - 10.4.1.2.8. Post Analytical Spike
  - 10.4.1.2.9. Parent Sample
  - 10.4.1.2.10. Serial Dilution
  - 10.4.1.2.11. MS
  - 10.4.1.2.12. MSD
  - 10.4.1.2.13. Maximum of 2 samples
  - 10.4.1.2.14. CCV2
  - 10.4.1.2.15. CCB2
  - 10.4.1.2.16. Maximum of 10 samples
  - 10.4.1.2.17. CCV3
  - 10.4.1.2.18. CCB3
- 10.4.1.3. Using the analytical sequence, arrange the digested standards and samples to be analyzed chronologically.
- 10.4.1.4. Transfer about 6 ml of the digestates into the autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence.
- 10.4.1.5. Prepare a Dilution Test sample at 5 times dilution. Pipette 2 ml of sample, add 8 ml of SO into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.
- 10.4.1.6. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS digestate.
- 10.4.1.7. Set the prepared samples into the autosampler and start the analytical run.

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**MERCURY IN LIQUID WASTE**SOP No.: EMAX-7470 Revision No. 7 Effective Date: 12-Dec-12**10.4.2. Sample Result Evaluation**

10.4.2.1. Check QC criteria as soon as data is available.

10.4.2.1.1. Check the LCS recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.

10.4.2.1.2. Check the MS, serial dilution and post-digestion spike<sup>1</sup> recovery against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.

10.4.2.1.3. Check sample result concentrations are within the calibration range.

10.4.2.2. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.2.2.1. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.2.2.2. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.2.3. Properly fill up the analytical run log.

10.4.2.4. Upload the electronic data to the network.

**10.4.3. Method of Standard Addition (MSA)**

10.4.3.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.3.2. Prepare three sample solutions (Ms1, Ms2, Ms3) to objectively produce equal increments of concentration in the final solution without diluting the sample more than 50% of its original volume and expected concentrations falls within the linear range.

*Example: Sample concentration is tentatively determined at 2 µg/L.**Ms1 – take 10 ml of digestate and add 0.2 ml of 100 µg/L spike standard (≈ 6 µg/L)**Ms2 – take 10 ml of digestate and add 0.4 ml of 100 µg/L spike standard (≈ 7 µg/L)**Ms3 – take 10 ml of digestate and add 0.6 ml of 100 µg/L spike standard (≈ 8 µg/L)*

10.4.3.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq.-10.5.7.

**10.5. Calculations****10.5.1. Calibration Factor**

$$CF = \frac{R_a}{C_a} \quad \text{Eq.-10.5.1}$$

where:

<sup>1</sup> This SOP defaults to Post-Digestion Spike recovery of 85-115% based on Method 7000A. However, if project specific requirements reference to Method 7000B, recovery requirement is 80 – 120%.

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- $CF$  – Calibration factor  
 $R_a$  – Response for analyte measured in absorbance  
 $C_a$  – Known concentration of analyte measured in  $\mu\text{g/L}$

10.5.2. Average Calibration Factor

$$ACF = \frac{\sum CF}{n} \quad \text{Eq.-10.5.2}$$

where:

- $ACF$  – Average calibration factor  
 $\sum CF$  – Sum of calibration factors  
 $n$  – Number of measurements

10.5.3. Correlation Coefficient

$$r(x, y) = \frac{\frac{1}{N} \sum_{i=1}^N (x_i - \bar{x})(y_i - \bar{y})}{(SD_x)(SD_y)} \quad \text{Eq.-10.5.3}$$

where:

- $r(x, y)$  – Correlation coefficient  
 $N$  – Number of measurements  
 $x_i$  – Found value of the  $i^{\text{th}}$  measurement  
 $\bar{x}$  – Mean of found values  
 $y_i$  – True value of the  $i^{\text{th}}$  measurement  
 $\bar{y}$  – Mean of true values  
 $SD_x$  – Standard deviation of the found values  
 $SD_y$  – Standard deviation of the true values

10.5.4. Sample Result

$$C = (R_s)(CF)(DF) \frac{V_e}{S_a} \quad \text{Eq.-10.5.4}$$

where:

- $C$  – Sample concentration in  $\mu\text{g/L}$   
 $CF$  – Calibration factor  
 $DF$  – Dilution factor  
 $R_s$  – Sample absorbance

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**MERCURY IN LIQUID WASTE**SOP No.: EMAX-7470 Revision No. 7 Effective Date: 12-Dec-12 $V_e$  – Extract volume in L $S_a$  – Sample amount (ml)10.5.5. Calculate for Percent Recovery

$$\% \text{ Recovery} = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.5.5}$$

where:

 $C_f$  – Concentration found $C$  – Concentration of the sample (use 0 for LCS) $C_s$  – Concentration of spike10.5.6. Calculate for Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.6}$$

where:

 $RPD$  – Relative Percent Difference $C_1$  – Measured concentration of the first sample aliquot $C_2$  – Measured concentration of the second sample aliquot10.5.7. Calculation for MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \quad \text{Eq.-10.5.7}$$

where:

 $C_x$  – Concentration of the sample $C_s$  – Concentration of spike $S_1$  – Analytical signal of MS1 $S_2$  – Analytical signal of MS2 $V_x$  – Volume of sample aliquot $V_s$  – Volume of spike or reagent water10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run and sample preparation log.

10.6.2. Print a copy of raw data and the QC report.

10.6.3. Highlight the data to be reported.

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10.6.4. Collate the reportable data separating the QC results from the sample results.

10.6.5. Keep all other data generated with the analytical folder marked with "For record only".

**10.7. Report Generation**

10.7.1. Generate the report using the following in-house reporting program:

Executable Files	Required Support Files	Output
WDB5.exe	Login File (requires network), project.pln, seq_name.hg	Method.txt [this file integrates the login sample information and the analytical sample information]
MSRB6.exe	method.txt, method.met, method.crf, project.pln, project code.txt, qcell.txt	Sample Results (Form1)
IQC3C.exe	method.txt, method.crf, method.qc, project.pln, project code.txt, qcell.txt	QC Summary for LCS, MS, Post Digestion Spike (Form 3)
CQ1.exe	method.txt, method.crf, method.qc, project.pln, project code.txt, qcell.txt	Summary for Dilution Test, Sample Duplicate (Form 3)
LABCHRN1.exe	method.txt	Lab Chronicle
CN2.exe	method.txt, Form 1	Case Narrative

**10.8. Data Review**

10.8.1. Arrange the analysis package in sequence as detailed below.

- 10.8.1.1. Case Narrative
- 10.8.1.2. Lab Chronicle
- 10.8.1.3. Sample Results
- 10.8.1.4. LCS/LCSD Summary
- 10.8.1.5. MS/MSD Summary
- 10.8.1.6. Post Digestion Spike Summary
- 10.8.1.7. Dilution Test Result Summary
- 10.8.1.8. Analytical Run Log
- 10.8.1.9. Raw Data
- 10.8.1.10. Sample Preparation Log
- 10.8.1.11. Non-Conformance Report (if any)

10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check method blank is compliant to Project Specific Requirements (PSR) criteria.

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- ✓ Check LCS/LCSD, MS/MSD and Dilution test against QC limits.
- ✓ Check analytical spike test if dilution test failed.
- ✓ Check for possible carry-over and if confirmation is performed.
- ✓ Review the attached logs that they are properly filled.
- ✓ Check the generated reports against the raw data, analytical run log and sample preparation log. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analytical folder for secondary review

**10.9. Preventive Maintenance**

10.9.1. Daily routine maintenance must be observed religiously. Observe manufacturer's notes regarding DOs and DON'Ts:

- System preparation is a ***MUST*** before instrument startup.
- Make certain that drying tube has been packed loosely. If drying tube is blocked, liquid may backflow into the optical cell; this will require disassembly and leaning.
- Do not shutdown the instrument when operational, abort the run first if interruption is needed.

10.9.2. Daily routine maintenance including checking of reductant solution, 10% SnCl<sub>2</sub>, trouble shooting and major repairs must be recorded in the maintenance log.

10.9.3. Maintain the instrument clean at all times.

10.9.4. For trouble shooting, consult the Operations Manual, Section 4.

**11.0 QUALITY CONTROL****11.1. Sample Preparation QC**

11.1.1. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06 for details.

11.1.2. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.1.3. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.

11.1.4. Every preparative batch must have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. Digest QC samples together with the field samples.

11.1.5. Properly treat all lab wares used in the sample preparations as specified in EMAX-QC07.

**11.2. Sample Analysis QC**

11.2.1. Every analytical run is preceded with an Initial Calibration (ICAL) and Initial Calibration Verification (ICV). The ICV standard is obtained from a different source from that of the initial calibration.

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Analyze an instrument calibration blank (ICB) after the ICV. No further analysis is valid unless acceptance criteria are met.

11.2.2. Verify calibration with Continuing Calibration Verification (CCV) standard and Continuing Calibration Blank (CCB) after every ten samples and at the end of the analytical run.

11.2.2.1. Perform Dilution Test whenever a new or unusual sample matrix is encountered.

11.2.2.2. Evaluate Post Digestion Spike result if the dilution test failed to meet the acceptance criteria.

11.2.3. Use Method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.4. Acceptance criteria, is summarized in Appendix 1.

**11.3. Method QC**

11.3.1. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response. The analytically determined concentration of this standard must be within 10% of the expected value.

11.3.2. A valid DL and LOD must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.3.3. All analysts conducting this analysis must have an established Demonstration of Capability (DOC) as described in EMAX-QA05.

**12.0 CORRECTIVE ACTION**

12.1. Corrective actions for each Quality Control Procedure is summarized in Appendix 1.

**12.2. Calibration**

12.2.1. Initial Calibration (ICAL) - if ICAL is non-compliant, consider the following suggestions to correct the problem:

- Replace the sample tubing, prepare fresh rinsate and re-prepare fresh SnCl<sub>2</sub>. Rinse the system for at least 15 minutes prior to calibration.
- If problem persist, run the latest calibration standard that passed to check for possible instrumentation problem. If it passes, this is an indication that no instrumentation problem exist, re-digest the calibration standards. If it failed, clean the lamp, prior to re-calibration.
- If problem persist, inform the supervisor for further action

12.2.2. Initial Calibration Verification (ICV) - if the ICV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest ICV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the ICV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.2.3. Continuing Calibration Verification (CCV) - If CCV is non-compliant, consider the following suggestions to correct the problem:

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- Run the latest CCV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the CCV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

**12.3. Sample Prep QC**

12.3.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.3.2. Method Blank (MB) - if MB is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.

**12.4. Sample Analysis QC**

12.4.1. Lab Control Sample (LCS) - If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution.
- Check instrument performance to determine if it is within acceptable guidelines.
- Re-calculate the data and/or re-analyze the extract if any of the above checks reveals a problem.
- If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate digestion problem, fill-up an NCR and order re-digestion to include the associated sample(s).

12.4.2. Matrix Spike (MS) - If MS is non-compliant, consider the following suggestions to correct the problem:

- If recovery failed to meet the acceptance criteria and sample result is > 5X the LOQ and the spike amount is > 4X the parent sample concentration, evaluate the post digestion spike sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.
- If recovery failed to meet the acceptance criteria and sample result is ~ 5X the LOQ and the spike amount is > 4X of the parent sample concentration, evaluate the serial dilution sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.

12.5. A Non-Conformance Report (NCR) is required when the following circumstances occur.

- Anomalies other than specified in Appendix 1, is observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08 for NCR details.

**13.0 POLLUTION PREVENTION**

13.1. Mercury is a very volatile element, dangerous levels are readily attained in air. Mercury vapour should not exceed 0.1 mg/m<sup>3</sup> in air. Air saturated with the vapor at 20°C contains mercury in a concentration far greater than that limit. The danger increases at higher temperatures. It is therefore important that mercury

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be handled with care. Containers of mercury must be securely covered and spillage must be avoided. Mercury must only be handled under in a well-ventilated area. Prepare all standards in the fume hoods.

- 13.2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. A bypass must be included on the system to vent the mercury vapor into an exhaust hood.
- 13.3. Small amounts of mercury spillage can be cleaned up by addition of sulphur powder. The resulting mixture must be properly labeled and turned over to the waste disposal unit for proper disposal.

**14.0 WASTE MANAGEMENT**

- 14.1. No samples may be dumped on the laboratory sink.
- 14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.
- 14.3. Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.
- 14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

- 15.1.1. Mercury – is also known as quicksilver, is a chemical (element) that occurs naturally in the environment in several forms. One form of mercury is used in thermometers. This form is called “metallic mercury”. Mercury is also used in barometers and other common consumer products. Mercury can also be combined with other chemicals, such as chlorine, carbon or oxygen to form either “inorganic” or “organic” mercury compounds.
- 15.1.2. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.1.3. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
  - 15.1.3.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
  - 15.1.3.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.4. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.1.5. Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

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- 15.1.6. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.1.7. Material Safety Data Sheet (MSDS) – is a written information concerning a chemical physical properties, toxicity, health standards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.1.8. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.9. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyze to monitor the accuracy of the analytical system.
- 15.1.13. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.15. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.17. Matrix – is a component or form of sample.
- 15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.20. Reagent Water – Purified water free from any target analyte or any other substances that may interfere with the analytical process.
- 15.2. **Application of EMAX QC Procedures**
- 15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Mercury analysis by Cold Vapor Absorption Technique. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.
- 15.3. **Department of Defense (DoD) Projects**

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15.3.1. Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

**15.4. Department of Energy (DoE) Projects**

15.4.1. Samples from DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, is applied.

**16.0 REFERENCES**

- 16.1. Method 7470A, Test Methods for Evaluating Solid Wastes, USEPA SW-846, 1992.  
 16.2. EMAX Quality Systems Manual, as updated.

**17.0 APPENDICES**

**17.1. Figures**

- 17.1.1. Figure 1 Autosampler Layout  
 17.1.2. Figure 2 Typical Calibration Curve  
 17.1.3. Figure 3 Typical Sample Result Summary  
 17.1.4. Figure 4 Typical LCS/LCSD Summary  
 17.1.5. Figure 5 Typical MS/MSD Summary  
 17.1.6. Figure 6 Typical Analytical Spike Summary  
 17.1.7. Figure 7 Typical Dilution Test Summary  
 17.1.8. Figure 6 Typical Case Narrative

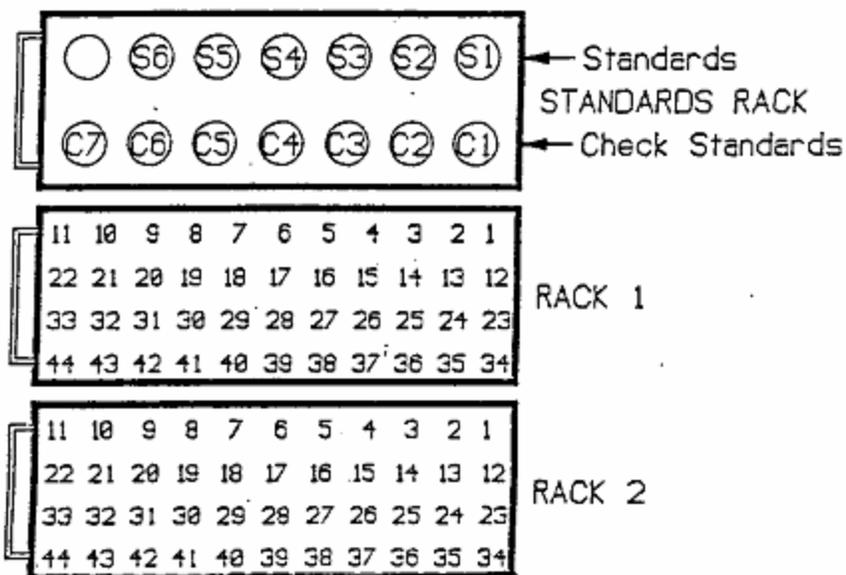
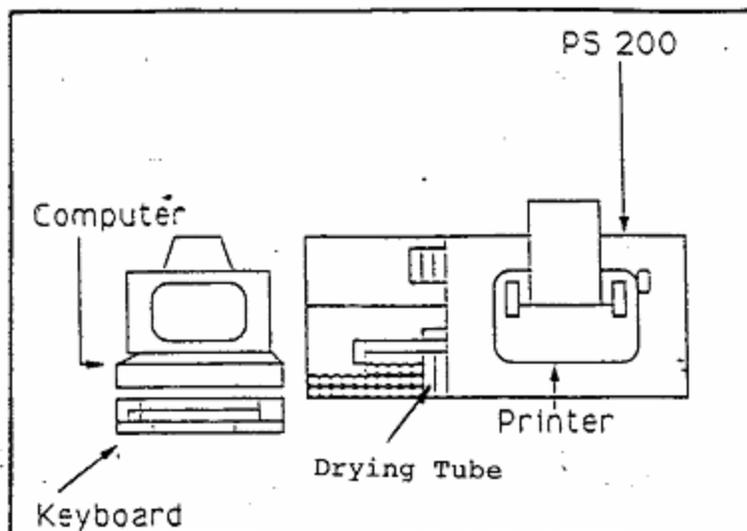
**17.2. Appendices**

- 17.2.1. Appendix 1 Summary of Quality Control Procedures  
 17.2.2. Appendix 2 Demonstration of Capability

**17.3. Forms**

- 17.3.1. 7470FS Sample Preparation Log  
 17.3.2. 7470FA Analytical Run Log  
 17.3.3. 7470FM Instrument Maintenance Log

Figure 1: AUTOSAMPLER LAYOUT



Autosampler layout

Figure 2:

TYPICAL CALIBRATION CURVE

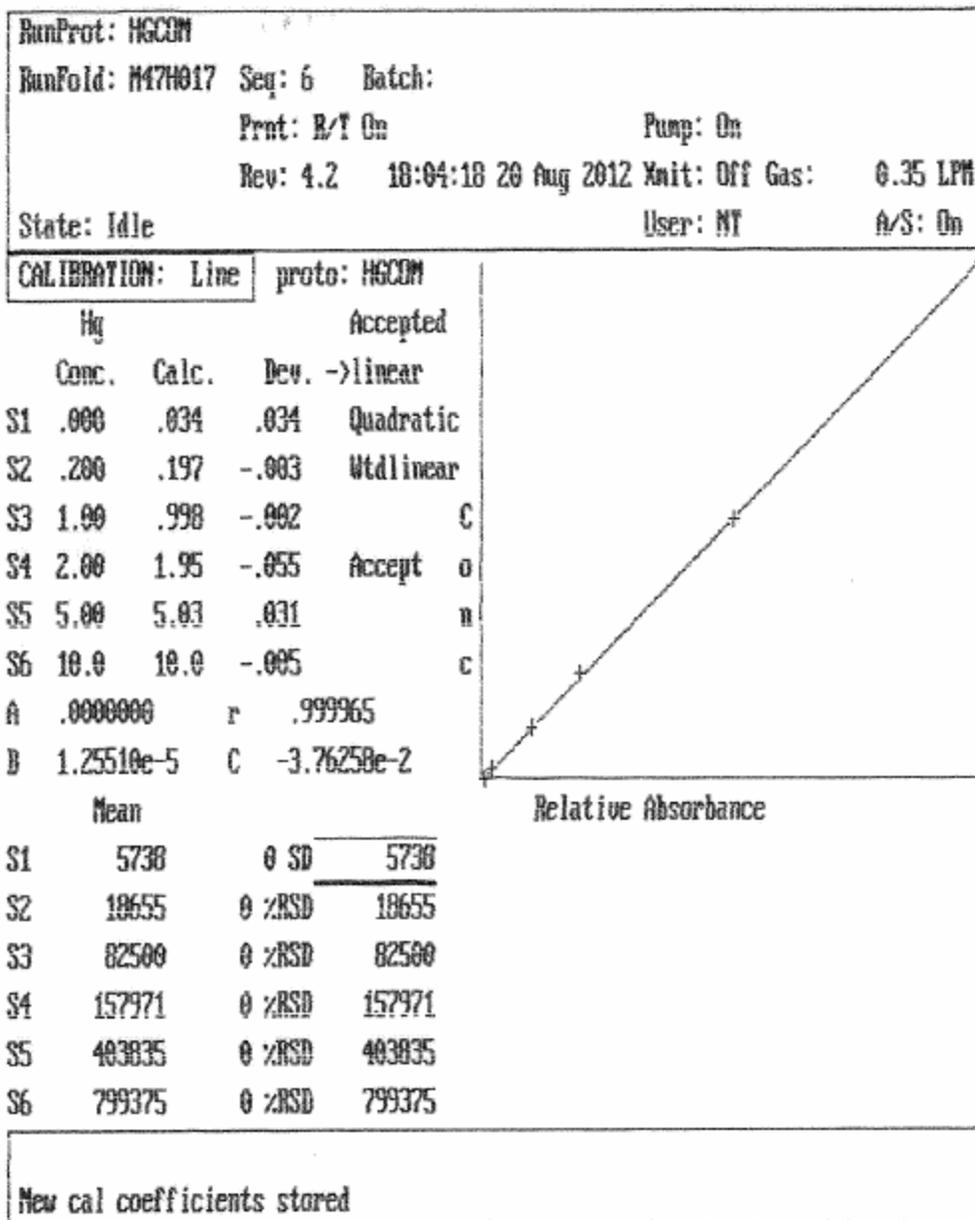


Figure 3:

TYPICAL SAMPLE RESULT SUMMARY

METHOD 7470A  
 MERCURY BY COLD VAPOR

Client : XYZ, INC.  
 Project : CLEAN WATER PROJECT  
 Batch No. : 12H108

Matrix : WATER  
 Instrument ID : TI047

SAMPLE ID	EMAX SAMPLE ID	RESULTS (ug/L)	DLF	MOIST	RL (ug/L)	MDL (ug/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK1W	HGH024WB	ND	1	NA	0.500	0.100	08/20/1218:15	08/20/1213:15	M47H017010	M47H017008	HGH024W	NA	08/20/12
LCS1W	HGH024WL	5.14	1	NA	0.500	0.100	08/20/1218:17	08/20/1213:15	M47H017011	M47H017008	HGH024W	NA	08/20/12
LCD1W	HGH024WC	5.14	1	NA	0.500	0.100	08/20/1218:19	08/20/1213:15	M47H017012	M47H017008	HGH024W	NA	08/20/12
MW-6AS	H108-05A	5.19	1	NA	0.500	0.100	08/20/1218:21	08/20/1213:15	M47H017013	M47H017008	HGH024W	08/16/12	08/16/12
MW-6	H108-05	ND	1	NA	0.500	0.100	08/20/1218:23	08/20/1213:15	M47H017014	M47H017008	HGH024W	08/16/12	08/16/12
MW-6DL	H108-05J	ND	5	NA	2.50	0.500	08/20/1218:25	08/20/1213:15	M47H017015	M47H017008	HGH024W	08/16/12	08/16/12
MW-6MS	H108-05M	5.43	1	NA	0.500	0.100	08/20/1218:27	08/20/1213:15	M47H017016	M47H017008	HGH024W	08/16/12	08/16/12
MW-6MSD	H108-05S	5.47	1	NA	0.500	0.100	08/20/1218:29	08/20/1213:15	M47H017017	M47H017008	HGH024W	08/16/12	08/16/12
Tt-MW33	H108-02	ND	1	NA	0.500	0.100	08/20/1218:31	08/20/1213:15	M47H017018	M47H017008	HGH024W	08/16/12	08/16/12
MW-7	H108-03	ND	1	NA	0.500	0.100	08/20/1218:33	08/20/1213:15	M47H017019	M47H017008	HGH024W	08/16/12	08/16/12
MW-5	H108-04	ND	1	NA	0.500	0.100	08/20/1218:40	08/20/1213:15	M47H017022	M47H017020	HGH024W	08/16/12	08/16/12
MW-3	H108-06	ND	1	NA	0.500	0.100	08/20/1218:42	08/20/1213:15	M47H017023	M47H017020	HGH024W	08/16/12	08/16/12
MW-2	H108-07	ND	1	NA	0.500	0.100	08/20/1218:44	08/20/1213:15	M47H017024	M47H017020	HGH024W	08/16/12	08/16/12
Tt-MW32	H108-09	ND	1	NA	0.500	0.100	08/20/1218:46	08/20/1213:15	M47H017025	M47H017020	HGH024W	08/16/12	08/16/12
Tt081612EB	H108-11	ND	1	NA	0.500	0.100	08/20/1218:50	08/20/1213:15	M47H017027	M47H017020	HGH024W	08/16/12	08/16/12
MW-1	H108-08	ND	1	NA	0.500	0.100	08/20/1220:29	08/20/1213:15	M47H017072	M47H017068	HGH024W	08/16/12	08/16/12
GMT-11	H108-10	ND	1	NA	0.500	0.100	08/21/1210:51	08/20/1213:15	M47H018010	M47H018008	HGH024W	08/16/12	08/16/12

**Figure 4: TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER PROJECT  
 SDG NO.: 12H108  
 METHOD: METHOD 7470A

MATRIX: WATER % MOISTURE: NA  
 DILTN FACTR: 1 1 1  
 SAMPLE ID: MBLK1W  
 CONTROL NO.: HGH024WB HGH024WL HGH024WC  
 LAB FILE ID: M47H017010 M47H017011 M47H017012  
 DATIME EXTRCTD: 08/20/1213:15 08/20/1213:15 08/20/1213:15 DATE COLLECTED: NA  
 DATIME ANALYZD: 08/20/1218:15 08/20/1218:17 08/20/1218:19 DATE RECEIVED: 08/20/12  
 PREP. BATCH: HGH024W HGH024W HGH024W  
 CALIB. REF: M47H017008 M47H017008 M47H017008

ACCESSION:

PARAMETER	BLNK RSLT ug/L	SPIKE AMT ug/L	BS RSLT ug/L	BS % REC	SPIKE AMT ug/L	BSD RSLT ug/L	BSD % REC	RPD %	QC LIMIT %	MAX RPD %
Mercury	ND	5	5.14	103	5	5.14	103	0	80-120	20

**Figure 5: TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER PROJECT  
 SDG NO.: 12H108  
 METHOD: METHOD 7470A

=====

MATRIX: WATER % MOISTURE: NA  
 DILTN FACTR: 1 1 1  
 SAMPLE ID: Mw-6  
 CONTROL NO.: H108-05 H108-05M H108-05S  
 LAB FILE ID: M47H017029 M47H017031 M47H017034  
 DATIME EXTRCTD: 08/20/1213:15 08/20/1213:15 08/20/1213:15 DATE COLLECTED: 08/16/12  
 DATIME ANALYZD: 08/20/1218:55 08/20/1219:00 08/20/1219:06 DATE RECEIVED: 08/16/12  
 PREP. BATCH: HGH024W HGH024W HGH024W  
 CALIB. REF: M47H017020 M47H017020 M47H017032

ACCESSION:

PARAMETER	SMPL RSLT ug/L	SPIKE AMT ug/L	MS RSLT ug/L	MS % REC	SPIKE AMT ug/L	MSD RSLT ug/L	MSD % REC	RPD %	QC LIMIT %	MAX RPD %
Mercury	ND	5	5.19	104	5	5.23	105	1	80-120	20

**Figure 6: TYPICAL ANALYTICAL SPIKE SUMMARY**

EMAX QUALITY CONTROL DATA  
 ANALYTICAL SPIKE ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER PROJECT  
 SDG NO.: 12H108  
 METHOD: METHOD 7470A

MATRIX: WATER % MOISTURE: NA  
 DILTN FACTR: 1 1  
 SAMPLE ID: MW-6  
 CONTROL NO.: H108-05 H108-05A  
 LAB FILE ID: M47H017014 M47H017013  
 DATIME EXTRCTD: 08/20/1213:15 08/20/1213:15 DATE COLLECTED: 08/16/12  
 DATIME ANALYZD: 08/20/1218:23 08/20/1218:21 DATE RECEIVED: 08/16/12  
 PREP. BATCH: HGH024W HGH024W  
 CALIB. REF: M47H017008 M47H017008

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	SPIKE AMT (ug/L)	AS RSLT (ug/L)	AS % REC	QC LIMIT ( % )
Mercury	ND	5	5.19	104	85-115

**Figure 7: TYPICAL DILUTION TEST SUMMARY**

EMAX QUALITY CONTROL DATA  
 SERIAL DILUTION ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER PROJECT  
 BATCH NO.: 12H108  
 METHOD: METHOD 7470A

MATRIX:	WATER		% MOISTURE:	NA
DILUTION FACTOR:	1	5		
SAMPLE ID:	MW-6	MW-6DL		
EMAX SAMP ID:	H108-05	H108-05J		
LAB FILE ID:	M47H017014	M47H017015		
DATE EXTRACTED:	08/20/1213:15	08/20/1213:15	DATE COLLECTED:	08/16/12
DATE ANALYZED:	08/20/1218:23	08/20/1218:25	DATE RECEIVED:	08/16/12
PREP. BATCH:	HGH024W	HGH024W		
CALIB. REF:	M47H017008	M47H017008		

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	SERIAL DIL RSLT (ug/L)	DIF RSLT %	QC LIMIT ( % )
-----	-----	-----	-----	-----
Mercury	ND	ND	0	10

**Figure 8: TYPICAL CASE NARRATIVE**

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN WATER PROJECT  
SDG : 12H108

METHOD 7470A  
MERCURY BY COLD VAPOR

A total of ten (10) water samples were received on 08/16/12 for Mercury analysis, Method 7470A in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

**Holding Time**  
Samples were analyzed within the prescribed holding time.

**Calibration**  
Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source. Continuing calibration verifications were carried out at the frequency specified by the project. All calibration requirements were within acceptance criteria.

**Method Blank**  
Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

**Lab Control Sample**  
A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for HGH024WL/C were all within QC limits.

**Matrix QC Sample**  
Matrix QC sample was analyzed at the frequency prescribed by the project. Percent recoveries for H108-05M/S were within project QC limits. In addition Analytical spike and serial dilution were analyzed for matrix interference evaluation. Results were within method acceptance criteria.

**Sample Analysis**  
Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURES**

PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Initial multipoint calibration	Daily initial calibration prior to sample analysis	Correlation coefficient $r \geq 0.995$ for linear regression	Correct the problem then repeat initial calibration		
Initial calibration verification (second source)	Daily after initial calibration	Analyte within $\pm 10\%$ of expected value	Correct the problem then repeat initial calibration		
Calibration verification (CCV)	Daily, before sample analysis, every 10 samples and at the end of analysis sequence	Analyte within $\pm 20\%$ of expected value	Repeat calibration and re-analyze all samples since last successful calibration		
Calibration blank (CCB)	After every calibration verification	No analyte detected $> LOD$	Correct the problem then re-analyze calibration blank and previous samples		
Method blank (MB)	One per preparation batch	No analyte detected $> \frac{1}{2} LOQ$	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
Lab Control Sample (LCS)	One LCS per preparation batch	%Rec.: 80-120%	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD or MS/Dup	One set MS/MSD or MS/Dup in every preparatory batch	%Rec. 80-120%, RPD $\leq 20\%$	Perform Post-Digestion Spike.		
Post-Digestion Spike	When MS/MSD or MS/Dup fails	%Rec. 85-115%	Perform dilution test if analyte concentration is sufficiently high ( $\sim 5x$ the LOQ after dilution), otherwise perform MSA.		
Dilution Test	When Post-Digestion Spike fails and analyte concentration is sufficiently high ( $\sim 5x$ the LOQ after dilution)	Within $\pm 10\%$ of the parent sample result	Perform MSA.		
Comments: Refer to PSR for flagging criteria LOQ = lowest calibration point			Reviewed By:		
			Date:		

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

DEMONSTRATION OF CAPABILITY  
 MERCURY  
 METHOD SW 7470

SOP: EMAX-7470  
 Conc Unit: mg/L  
 Sample Amount(mL): 50

Extraction dates: 12/21/2011  
 Analysis dates: 12/21/2011  
 Extracted and Analyzed by: N. Tan

PARAMETER	HGL013WL	HGL013WC	HGL014WL	HGL014WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	M47L014-11	M47L014-12	M47L014-61	M47L014-62							
Mercury	5.18	5.11	5.13	5.17	5	5.15	103	0.033	1	80 - 120	PASSED







SOP REVIEW FORM

EMAX-7471

Rev. 7

MERCURY IN SOLID OR SEMISOLID WASTE

SOP No.

Revision Number

Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read the following MSDS:
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

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This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

Mary Jane Mendoza Jm

Date:

7/8/14

## STANDARD OPERATING PROCEDURE

**MERCURY IN SOLID OR SEMISOLID WASTE**

SOP No.: EMAX-7471 Revision No. 7 Effective Date: 12-Dec-12

Prepared By: Mary Jane Mendoza Date: 12.11.12

Approved By: Kenette Pimentel *[Signature]* Date: 12.11.12  
QA Manager

Approved By: Caspar Pang *[Signature]* Date: 12-11-12  
Laboratory Director

Control Number: 7471-07-

**1.0 SCOPE AND APPLICATION**

- 1.1. This procedure applies to the measurement of Mercury in domestic and industrial wastes, soils, sediments, extracts and sludge samples by Cold Vapor Absorption Technique.
- 1.2. This SOP is an adaptation of Method 7471B.

**2.0 SUMMARY OF METHOD**

- 2.1. A representative amount of sample is digested in nitric and hydrochloric acids, followed by oxidation with potassium permanganate.
- 2.2. Organic mercurial are broken down and converted into mercuric ions in order to respond to the cold vapor atomic absorption technique. Addition of permanganate ensures that organo-mercury compounds are oxidized.
- 2.3. Absorption of radiation by mercury vapor at 253.7 nm is then measured in the digested samples.
- 2.4. **Interferences**
  - 2.4.1. Sulfides (as sodium sulfide), copper and chloride at high concentrations are known to interfere with the recovery of mercury. Samples containing such interference may require additional permanganate (about 12.5 ml).
  - 2.4.2. Care must be taken to ensure that free chlorine is absent before the mercury is swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent. In addition, the dead air space in the digestion vessel must be purged before adding stannous sulfate.
  - 2.4.3. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

**3.0 DETECTION LIMITS****3.1. Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

- 3.1.1. Refer to EMAX-QA04 for validation, verification and generation of DL, LOD and LOQ.
- 3.1.2. Established DL, LOD and LOQ are:

MATRIX	DL	LOD	LOQ
Soil (mg/Kg)	0.01	0.02	0.1

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**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (See Section 9.6). All samples analyzed above this range are considered "over-range" and requires dilution to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range are considered "under-range" and requires lower dilution factor to properly quantitate.

**5.0 SAMPLE HOLDING TIME AND PRESERVATION****5.1. Sample Collection**

- 5.1.1. Samples are expected to be contained in a jar or Shelby tube and cooled to  $\leq 6^{\circ}\text{C}$  without freezing.

**5.2. Holding Time**

- 5.2.1. Digest all samples within 28 days from date of collection.

**5.3. Preservation**

- 5.3.1. Store the samples at  $\leq 6^{\circ}\text{C}$  without freezing.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow Review
- 6.2. EMAX-QA04 Detection Limit (DL)
- 6.3. EMAX-QA05 Training
- 6.4. EMAX-QA08 Corrective Action
- 6.5. EMAX-QC01 Quality Control of Chemicals
- 6.6. EMAX-QC02 Analytical Standard Preparation
- 6.7. EMAX-QC06 Calibration of Micropipettes
- 6.8. EMAX-QC07 Glassware Cleaning
- 6.9. EMAX-SM03 Waste Disposal
- 6.10. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves, at all times when performing this procedure. Perform preparation and analysis of mercury in a fume hood equipped with an exhaust fan or blower.

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- 7.3. If for any reason, sample and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritation persists inform your supervisor immediately so that proper action can be taken.
- 7.4. Do not look directly at the Mercury Lamp while lit. The radiation may cause damage to your eyes.
- 7.5. Perform all reagent additions under a fume hood.
- 7.6. Mercury analyzers are to be used by trained personnel only.

**8.0 INSTRUMENTS, CHEMICALS AND REAGENTS****8.1. Instruments and Supplies**

## 8.1.1. Mercury Analyzers

8.1.1.1. Leeman PS-200 Automated Mercury Analyzer with Autosampler, Computer, Printer and PS 200 Software.

8.1.1.2. Leeman Hydra AA Automated Analyzer with Autosampler, Computer, Printer and PS200 Software.

8.1.2. 100 ml Digestion vessel

8.1.3. Digestate containers

8.1.4. Digestion block or equivalent.

8.1.5. Magnetic stirrer

8.1.6. Micropipettes and tips

8.1.7. Thermometer

**8.2. Chemicals and Reagents**

8.2.1. Hydrochloric acid, concentrated. Reagent grade.

8.2.2. Nitric acid, concentrated. Reagent grade.

8.2.3. Stannous Chloride: Dissolve 200 g. of SnCl<sub>2</sub> in reagent water, add 200 ml concentrated HCl and dilute to 2 L.

8.2.4. Hydroxylamine hydrochloride, 12% solution: Dissolve 120 g of hydroxylamine hydrochloride in 1 L reagent water.

8.2.5. Potassium permanganate, 5% solution. Dissolve 50 g of potassium permanganate in 1 L reagent water.

8.2.6. Aqua Regia – 1 part HNO<sub>3</sub> : 3 parts HCl solution

8.2.7. Reagent water – mercury-free water

8.2.8. Silica Sand for blank soil matrix

**9.0 STANDARDS**

- 9.1. Refer to EMAX-QC02 for proper analytical standard preparation.

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9.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.3. **Stock Standard**

9.3.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.

9.3.2. Transfer standards on a properly labeled inert vial with minimal headspace and store at -10°C to -20°C.

9.3.3. Prepare calibration standards from the primary standard.

9.3.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

Stock Std	Name	Source	CAT #	Conc.	Notes
Primary / CCV	Mercury	Leeman	602-00064	100 mg/L	Or equivalent
ICV/LCS/MS	Mercury	ERA	027	1000 mg/L	Or equivalent

9.4. **Intermediate Standard Solution**

9.4.1. From 100 mg/L stock solution take a 2 ml aliquot and dilute to 200 ml using reagent water. The solution shall have a final concentration of 1.0 mg/L.

9.4.2. Prepare secondary dilution from 1000 mg/L stock solution, take a 1 ml aliquot and dilute to 100 ml using reagent water. This solution shall have a final concentration of 10 mg/L.

9.5. **Working Standard**

9.5.1. From the secondary dilution of intermediate standard, prepare the working standard solution to have a final concentration of 100 µg/L.

9.6. **Initial Calibration Standards**

9.6.1. From the working solution, prepare the following *Leeman* standards.

Level	Aliquot (ml)	Final Digestion Volume (ml)	Concentration (µg/L)
S1	0	100	0
S2	0.2	100	0.2
S3	1.0	100	1.0
S4	2.0	100	2.0
S5	5.0	100	5.0
S6	10.0	100	10.0
CCV	5.0	100	5.0

9.7. **ICV/ LCS/MS**

9.7.1. From the working standard, prepare ICV/CCV/LCS/MS solutions using *ERA* Standards.

Name	Aliquot (ml)	Final Digestion Volume (ml)	Concentration (µg/L)
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ICV	2.0	100	2.0
LCS/MS	5.0	100	5.0

**10.0 PROCEDURES****10.1. Sample Preparation**

- 10.1.1. Weigh a representative 0.6 g portion of wet sample into digestion vessels.
- 10.1.2. Add spike standards to the LCS/MS. Add appropriate standards for Initial Calibration, ICV and CCVs in clean digestion vessels. (Refer to Section 9.6.1 and 9.7.1 for spike amounts).
- 10.1.3. Add 5 ml of reagent water and 5 ml aqua regia with mixing after each addition.
- 10.1.4. Heat for 2 minutes at  $95^{\circ}\text{C} \pm 3^{\circ}\text{C}$  in the digestion block, cool and add 50 ml of reagent water.
- 10.1.5. Add 15 ml of 5%  $\text{KMnO}_4$  solution to each bottle.
- 10.1.6. Swirl each vessel to mix and let it stand for 15 mins. Check each vessel if purple color persists. If not, add permanganate solution at 2.5 ml increments swirling the digestion vessel at every addition until purple color persists.
- Add the maximum amount of permanganate solution added to a solution, to the method blank, LCS, calibration standards and calibration verification standards.*
- 10.1.7. Place samples on the digestion block for 30 minutes at  $95^{\circ}\text{C} \pm 3^{\circ}\text{C}$ .
- 10.1.8. Allow the samples to cool.
- 10.1.9. Add 6 ml hydroxylamine hydrochloride solution and dilute to 100 ml using reagent water.
- 10.1.10. Properly fill up the Sample Preparation Log.

**10.2. Instrument Parameters****10.2.1. PROTOCOL****10.2.1.1. Set values as follows.**

Instrument ID:	PS200	HYDRA AA
Number of Integration	1	
Uptake time	20 sec.	18 sec.
Weight	N	N
Dilution	N	N
On/Off, times, gains		
On	Y	Y
Time	10	10
Gas	0.35 LPM	0.15 LPM

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Pump Rate	5 ml/min	7 ml/min
AUTOSAMPLER – Setup		
Station 1 (rack1)	From cup 1 to cup 44	From cup 1 to cup 44
Station 2 (rack 2)	From cup 1 to cup 44	From cup 1 to cup 44
Rinse time	60 sec.	60 sec.
CALIBRATION	Concentration, µg/L	Concentration, µg/L
S1, S2, S3, S4, S5, S6	0 0,.20, 1.0, 2.0, 5.0, 10.0	0 0,.20, 1.0, 2.0, 5.0, 10.0

## 10.2.2. DATA OUTPUT – Specify Report

Data Output	Real Time	Post Run
Samples	Y	Y
Standards	Y	Y
Updates	Y	Y
Peaks	N	N
IEC Stds.	N	N
Check Stds.	Y	Y
Dups and % Diff.	Y	Y
Wavelength	N	N
Rel. Absorbances	N	N
% RSD	Y	Y
Scans to PRN		N
Detail		Y
Summary		N
Post Run Copies		1
Post Run Report Order [ 1-sorted; 2- sequential]	2	

## 10.3. Calibration

10.3.1. Instrument Set-up

- 10.3.1.1. Set up PS200 or Hydra AA to proper operating parameters. Refer to Section 10.2.1.  
*New pump tubing, must ran with rinse for 45 minutes to break in the tubing.*
- 10.3.1.2. Turn on lamp and allow to warm up for at least 5 minutes.
- 10.3.1.3. Check the peristaltic pump to deliver a steady flow.

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10.3.1.4. Check that the reductant solution, 10% SnCl<sub>2</sub>, is sufficient. If not, prepare solution as described in Section 8.2.3.

10.3.2. Initial Calibration (ICAL)

10.3.2.1. Prepare initial calibration solution as described in Section 9.6.1.

10.3.2.2. Perform the same procedure used for analytical samples as described in Section 10.1.

10.3.2.3. Analyze as described in Section 10.4.

10.3.2.4. Refer to Section 10.5 for calculations.

10.3.2.5. Initiate initial calibration as described in the instrument operations manual and acquire the calibration data for review after calibration is completed.

10.3.2.6. Refer to Appendix 1 for Quality Control acceptance criteria.

10.3.2.7. Verify the initial calibration by a secondary source standard.

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Prepare ICV as described in Section 9.7.

10.3.3.2. Perform the same procedure used for analytical samples as described in Section 10.1.

10.3.3.3. Analyze the ICV sample to verify the concentration of the ICAL.

10.3.3.4. Refer to Appendix 1 for Quality Control acceptance criteria.

10.3.4. Continuing Calibration Verification (CCV)

10.3.4.1. Prepare CCV as described in Section 9.6.

10.3.4.2. Perform the same procedure used for analytical samples as described in Section 10.1.

10.3.4.3. Analyze the CCV sample to verify the concentration of the ICAL.

10.3.4.4. Refer to Appendix 1 for Quality Control acceptance criteria.

10.4. **Analysis**10.4.1. Calibration

10.4.1.1. Refer to the instrument operations manual for proper calibration and analytical sequence setup (autosampler setup).

10.4.1.2. Analytical batch ID naming convention: MIIMSSS

where:

M – is for Mercury and is always the first character

II – is the instrument number

M – is the month code (A for January, B for February, and so on)

SSS – is a sequential number (resets to 001 for the first folder created each month)

10.4.1.3. **Typical Calibration Sequence**

S1	0.00000
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S2	0.20000
S3	1.00000
S4	2.00000
S5	5.00000
S6	10.0000

10.4.2. Analytical Sequence

- 10.4.2.1. ICV
  - 10.4.2.2. ICB
  - 10.4.2.3. CCV1
  - 10.4.2.4. CCB1
  - 10.4.2.5. Method Blank (MB)
  - 10.4.2.6. Lab Control Sample (LCS)
  - 10.4.2.7. Lab Control Sample Duplicate (LCSD)
  - 10.4.2.8. Post Analytical Spike
  - 10.4.2.9. Parent Sample
  - 10.4.2.10. Serial Dilution
  - 10.4.2.11. Matrix Spike (MS)
  - 10.4.2.12. Matrix Spike Duplicate (MSD)
  - 10.4.2.13. Maximum of 2 samples
  - 10.4.2.14. CCV2
  - 10.4.2.15. CCB2
  - 10.4.2.16. Maximum of 10 samples
  - 10.4.2.17. CCV3
  - 10.4.2.18. CCB3
- 10.4.3. Prepare a Dilution Test sample at 5x dilution. Pipette 2 ml of sample, add 10 ml of S<sub>0</sub> into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.
- 10.4.4. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.
- 10.4.5. Check QC criteria as soon as the data is available.
- 10.4.5.1. Check the LCS recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.
  - 10.4.5.2. Check the matrix spike recovery against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.

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10.4.5.3. Check that sample result concentrations are within the calibration range.

10.4.6. Dealing with Carryover

10.4.6.1. Check the sample analyzed preceded by another sample found to have target analyte concentrations exceeding the calibration range.

10.4.6.2. If no target analyte is detected as found in preceding high concentration sample, proceed with data reduction.

10.4.6.3. If there is any target analyte detected as found in preceding high concentration sample, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.7. Method of Standard Addition (MSA)

10.4.7.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.7.2. Prepare three sample solutions (Ms1, Ms2, Ms3) to objectively produce equal increments of concentration in the final solution without diluting the sample more than 50% of its original volume and expected concentrations falls within the linear range.

*Example: Sample concentration is tentatively determined at 2 µg/L.*

*Ms1 – take 10 ml of digestate and add 0.2 ml of 100 µg/L spike standard (≈ 6 µg/L)*

*Ms2 – take 10 ml of digestate and add 0.4 ml of 100 µg/L spike standard (≈ 7 µg/L)*

*Ms3 – take 10 ml of digestate and add 0.6 ml of 100 µg/L spike standard (≈ 8 µg/L)*

10.4.7.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq.-10.5.7.

10.4.7.4. Upload the electronic data to the network.

10.4.8. Sample Result Evaluation

10.4.8.1. Check that the analytical data generated which indicates positive results are quantitatively correct.

10.4.8.2. Check that analytical results are generated by the prescribed calibration schedule of method.

10.4.8.3. Check for possible carry-over. Re-analyze samples having trace level concentration preceded by a sample having a concentration over the calibration range.

10.4.8.4. Check the MS/MSD, serial dilution and the post digestion spike<sup>1</sup> results. If a matrix interference is indicated, check the PSR if MSA is waived. Otherwise, refer to Section 10.4.7.

10.4.8.5. Properly fill up the Analytical Run Log.

10.5. **Calculations**

10.5.1. Calibration Factor (CF)

<sup>1</sup> This SOP defaults to Post-Digestion Spike recovery of 85-115% based on Method 7000A. However, if project specific requirements reference to Method 7000B, recovery requirement is 80-120%.

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$$CF = \frac{R_a}{C_a} \quad \text{Eq.-10.5.1}$$

where:

- $R_a$  – Response for analyte measured in absorbance  
 $C_a$  – Known concentration of analyte measured in  $\mu\text{g/L}$

10.5.2. Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF}{n} \quad \text{Eq.-10.5.2}$$

where:

- $\sum CF$  – Sum of calibration factors  
 $N$  – Number of calibration factors

10.5.3. Correlation Coefficient

$$r(x, y) = \frac{\frac{1}{N} \sum_{i=1}^N (x_i - \bar{x})(y_i - \bar{y})}{(SD_x)(SD_y)} \quad \text{Eq.-10.5.3}$$

where:

- $r(x, y)$  – Correlation coefficient  
 $N$  – Number of measurements  
 $X_i$  – Found value of the  $i^{\text{th}}$  measurement  
 $\bar{x}$  – Mean of found values  
 $Y_i$  – True value of the  $i^{\text{th}}$  measurement  
 $\bar{y}$  – Mean of true values  
 $SD_x$  – Standard deviation of the found values  
 $SD_y$  – Standard deviation of the true values

10.5.4. Sample Result

$$C = (R_s)(CF)(DF) \frac{V_e}{S_a} \quad \text{Eq.-10.5.4}$$

where:

- $C$  – Sample concentration in  $\mu\text{g/L}$  or  $\mu\text{g/Kg}$   
 $CF$  – Calibration factor  
 $DF$  – Dilution factor  
 $R_s$  – Sample absorbance

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**MERCURY IN SOLID OR SEMISOLID WASTE**SOP No.: EMAX-7471 Revision No. 7 Effective Date: 12-Dec-12 $V_e$  – Extract volume in L $S_a$  – Sample amount in ml or g10.5.5. Calculate for Percent Recovery

$$\% \text{ Recovery} = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.5.5}$$

*where:* $C_f$  – Concentration found $C$  – Concentration of the sample (use 0 for LCS) $C_s$  – Concentration of spike10.5.6. Calculate for Percent Recovery

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.6}$$

*where:* $RPD$  – Relative Percent Difference $C_1$  – Measured concentration of the first sample aliquot $C_2$  – Measured concentration of the second sample aliquot10.5.7. Calculation for MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \quad \text{Eq.-10.5.7}$$

*where:* $C_x$  – Concentration of the sample $C_s$  – Concentration of spike $S_1$  – Analytical signal of MS1 $S_2$  – Analytical signal of MS2 $V_x$  – Volume of sample aliquot $V_s$  – Volume of spike or reagent water10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run log and sample preparation log.

10.6.2. Print a copy of the raw data, the QC report and the digestion log.

10.6.3. Highlight the data to be reported.

10.7. **Report Generation**

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10.7.1. Generate the report using the following in-house reporting program:

Executable Files	Required Support Files	Output
WDBX <sup>2</sup> .exe	Login File (requires network) Seq_name.sq; Gcints.txt	Method.txt [this file integrates the login sample information and the analytical sample information]
MSRBX.exe	Method.txt; Method.met; Method.crf; Project.pln; Qcell.txt	Sample Results (Form1)
IQCVXC.exe	Method.txt; Method.crf; Method.qc; Project.pln;Qcell.txt	QC Summary for LCS, MS, Post Digestion Spike
CQX.exe	Method.txt; Method.crf; Method.qc; Project.pln;Qcell.txt	Summary for Dilution Test, Sample Duplicate
LABCHRNX.exe	Method.txt	Lab Chronicle
CN2.exe		Case Narrative

**10.8. Data Review**

10.8.1. Arrange the analysis package in sequence as detailed below:

- 10.8.1.1. Case Narrative
- 10.8.1.2. Lab Chronicle
- 10.8.1.3. Sample Results
- 10.8.1.4. LCS/LCSD Summary
- 10.8.1.5. MS/MSD Summary
- 10.8.1.6. Dilution Test Report Summary
- 10.8.1.7. Post Digestion Spike Summary
- 10.8.1.8. Analytical Run Log
- 10.8.1.9. Raw Data
- 10.8.1.10. Sample Preparation Log
- 10.8.1.11. Non-Conformance Report (if any)

10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check the Method Blank is compliant to Project Specific Requirement (PSR) criteria.
- ✓ Check LCS/LCSD, MS/MSD and dilution test against the PSR. In the absence of the PSR, default to in-house QC limits.
- ✓ Evaluate the analytical spike test if dilution test failed.
- ✓ Check for possible carry-over and if so, check if confirmation is performed.
- ✓ Review the attached sample preparation log and analytical run log are properly filled.

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<sup>2</sup> X – latest program version

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- ✓ Check the generated reports against the raw data, analytical run log and digestion log. Check the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analytical folder for secondary review.

**10.9. Preventive Maintenance**

10.9.1. Daily routine maintenance shall be observed religiously. Observe manufacturer's notes regarding Dos and DONTs:

- System preparation is a ***MUST*** before instrument startup.
- Make certain that drying tube has been packed loosely. If drying tube is blocked, liquid may backflow into the optical cell. This will require disassembly and cleaning.
- Do not shutdown the instrument when operational. Abort the run first if interruption is needed.

10.9.2. Record daily routine maintenance, troubleshooting and major repairs in the maintenance log.

10.9.3. Maintain the instrument clean at all times.

10.9.4. For troubleshooting, consult the Operations Manual, Section 4.

**11.0 QUALITY CONTROL****11.1. Sample Preparation QC**

11.1.1. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lot of reagents.

11.1.2. Every preparative batch must have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples is digested together with the field samples.

11.1.3. All reagents are subjected to QC check prior to use. Refer to EMAX-QC01.

**11.2. Sample Analysis QC**

11.2.1. Every analytical run are preceded by an initial calibration and an initial calibration verification. The ICV standard should be obtained from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis are valid unless acceptance criteria are met.

11.2.2. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run.

11.2.2.1. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.2.2. Evaluate Post Digestion Spike result if the dilution test failed to meet the acceptance criteria.

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11.2.3. Use Method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.4. Refer to Appendix 1 for acceptance criteria.

**11.3. Method QC**

11.3.1. Detection Limit must be established before the analytical procedure can be used and a quarterly verification must be performed.

11.3.2. Method proficiency must be established before the analytical procedure can be used.

11.3.3. All analysts conducting this analysis must have established demonstration of capability

11.3.4. Perform Instrument Detection Limit quarterly unless otherwise specified by the project.

**12.0 CORRECTIVE ACTION****12.1. Calibration**

12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

- Replace the sample tubing, prepare fresh rinsate and re-prepare fresh SnCl<sub>2</sub>. Rinse the system for at least 15 minutes prior to calibration.
- If problem persists, run the latest passing calibration standard to check for possible instrumentation problem. If it passes, this is an indication that no instrumentation problem exists. Re-digest the calibration standards. If it fails, clean the lamp prior to re-calibration.
- If problem persists, inform the supervisor for further action.

12.1.2. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest passing ICV standard to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the ICV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.1.3. If the CCV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest passing CCV standard to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the CCV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

**12.2. Sample Prep QCs**

12.2.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM. Otherwise, perform the corrective action as specified in the PSR.

12.2.2. If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution.
- Check instrument performance to determine if it is within acceptable guidelines.
- Re-calculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

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- If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate digestion problem, fill-up an NCR and order re-digestion to include the associated sample(s).

12.2.3. If MS is non-compliant, consider the following suggestions to correct the problem:

- If recovery failed to meet the acceptance criteria, and sample result is > 5X the LOQ, and the spike amount is > 4X of the parent sample concentration, evaluate the serial dilution sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.
- If recovery failed to meet the acceptance criteria, and sample result is ≤ 5X the LOQ, and the spike amount is > 4X the parent sample concentration, evaluate the post digestion spike sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.

**13.0 POLLUTION PREVENTION**

- 13.1. Mercury is a very volatile element, dangerous levels are readily attained in air. Mercury vapor should not exceed 0.1 mg/m<sup>-3</sup> in air. Air saturated with the vapor at 20°C contains mercury in a concentration far greater than that limit. The danger increases at higher temperatures. It is, therefore, important that mercury be handled with care. Containers of mercury should be securely covered and spillage should be avoided. Mercury should only be handled under the hood in a well-ventilated area. Prepare all standards in the fume hoods.
- 13.2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. A by-pass must be included on the system to vent the mercury vapor into an exhaust hood.
- 13.3. Small amounts of mercury spillage can be cleaned up by addition of sulfur powder. The resulting mixture should be properly labeled and turned over to the waste disposal unit for proper disposal.

**14.0 WASTE MANAGEMENT**

- 14.1. No samples may be dumped on the laboratory sink.
- 14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.
- 14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.
- 14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES**

- 15.1. Since there is no technical difference between SW846 7471A and 7471B, this SOP may also be applicable for projects requiring method SW846 7471A.

**15.2. Definition of Terms**

- 15.2.1. Mercury – Also known as quicksilver, is a chemical (element) that occurs naturally in the environment in several forms. One form of mercury is used in thermometers. This form is called

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“metallic mercury”. Mercury is also used in barometers and other common consumer products. Mercury can also be combined with other chemicals, such as chlorine, carbon or oxygen to form either “inorganic” or “organic” mercury compounds.

- 15.2.2. **Analyte** – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.2.3. **Batch** – Is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.2.3.1. **Preparation batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.2.3.2. **Analytical batch** – is composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.2.4. **Detection Limit (DL)** - is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.2.5. **Limit of Detection (LOD)** - is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.
- 15.2.6. **Limit of Quantitation (LOQ)** - is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.2.7. **Material Safety Data Sheet (MSDS)** – is a written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.2.8. **Calibration** – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.2.9. **Calibration Blank** – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.2.10. **Instrument Method** – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.2.11. **Method Blank** – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.2.12. **Lab Control Sample (LCS)** – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.2.13. **Lab Control Sample Duplicate (LCSD)** – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.2.14. **Sample** – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID

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are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

- 15.2.15. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.2.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.2.17. Matrix – is a component or form of a sample.
- 15.2.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.2.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.2.20. Corrective Action – Action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 15.2.21. Non-conformance – An indication or judgment that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.
- 15.2.22. Raw Data – Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheet, record, memoranda, notes or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

### 15.3. **Application of EMAX QC Procedures**

- 15.3.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing analysis for mercury. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

### 15.4. **Department of Defense (DoD) Projects**

- 15.4.1. Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update is applied.

### 15.5. **Department of Energy (DoE) Projects**

- 15.5.1. Samples from DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update is applied.

## 16.0 **REFERENCES**

- 16.1. Method 7471B, Rev. 2, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, USEPA SW-846, Feb. 2007.
- 16.2. EMAX Quality Systems Manual, as updated.

## STANDARD OPERATING PROCEDURE

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**17.0 APPENDICES****17.1. Figures**

- 17.1.1. Figure 1 Autosampler Layout
- 17.1.2. Figure 2 Typical Calibration Curve
- 17.1.3. Figure 3 Typical Sample Result Summary
- 17.1.4. Figure 4 Typical LCS/LCSD Summary
- 17.1.5. Figure 5 Typical MS/MSD Summary
- 17.1.6. Figure 6 Typical Dilution Test Report Summary
- 17.1.7. Figure 7 Typical Post Digestion Spike Summary
- 17.1.8. Figure 8 Typical Case Narrative

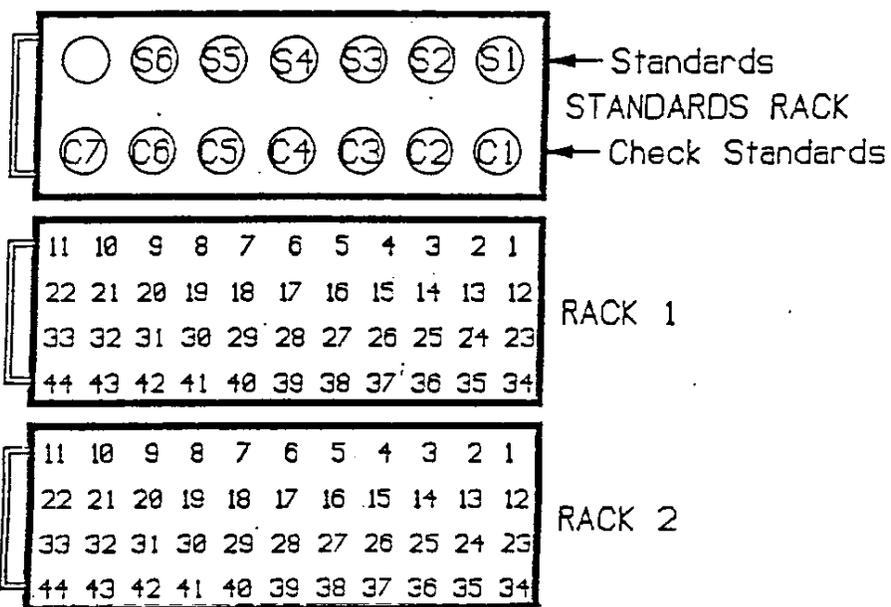
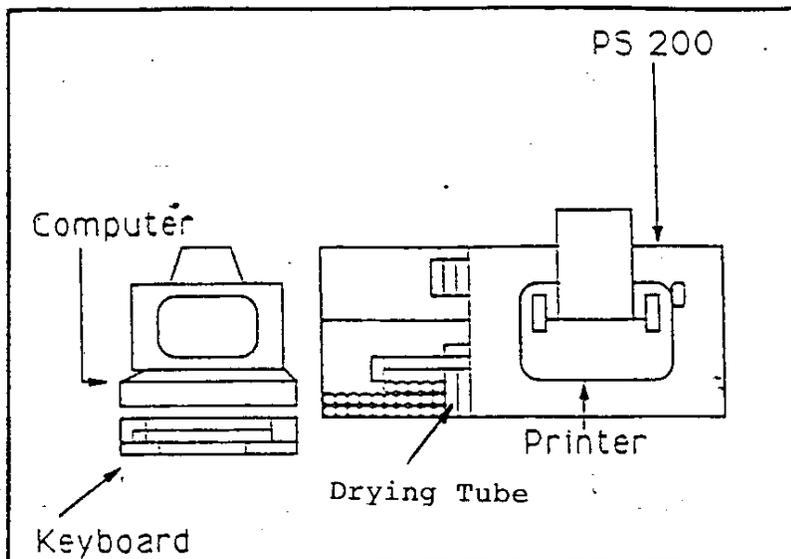
**17.2. Appendices**

- 17.2.1. Appendix 1 Summary of Quality Control Procedures
- 17.2.2. Appendix 2 Demonstration of Capability

**17.3. Forms**

- 17.3.1. 7471FS Sample Preparation Log
- 17.3.2. 7471FA Analytical Run Log
- 17.3.3. 7471FM Instrument Maintenance Log

Figure 1: AUTOSAMPLER LAYOUT

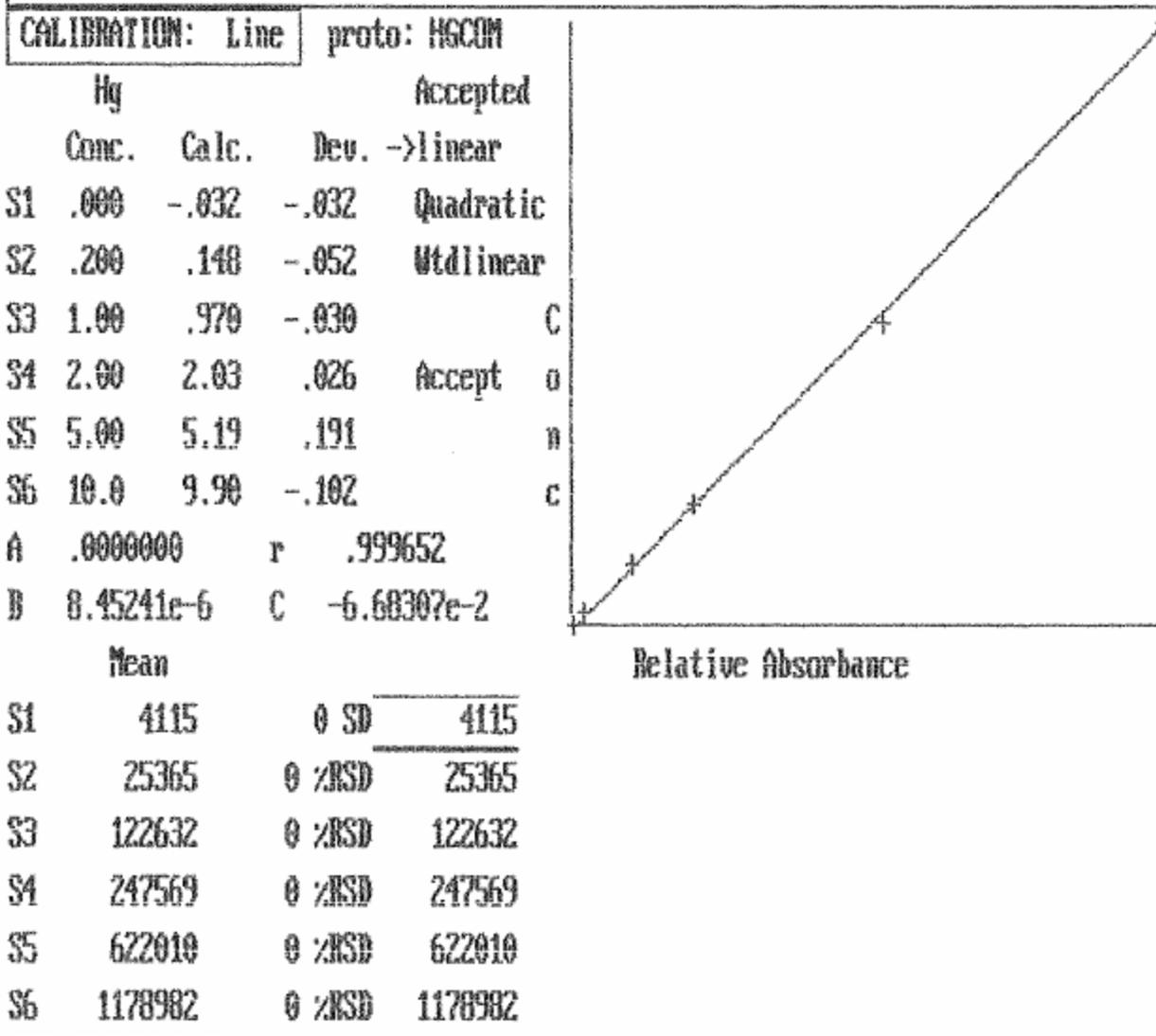


Autosampler layout

Figure 2: TYPICAL CALIBRATION CURVE

```

RunProt: HGCOM
RunFold: M47C011  Seq: 6  Batch:
                Prnt: R/T On                Pump: On
                Rev: 4.2  15:23:19 12 Mar 2010 Xmit: Off Gas: 0.35 LPM
State: Idle                User: NT/JC        A/S: On
    
```



New cal coefficients stored

Figure 3: TYPICAL SAMPLE RESULT SUMMARY

METHOD 7471B  
 MERCURY BY COLD VAPOR

=====  
 Client : XYZ, INC. Matrix : SOIL  
 Project : CLEAN LAND PROJECT Instrument ID : TI047  
 Batch No. : 10C029  
 =====

SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/kg)	DLF	MOIST	LOQ (mg/kg)	LOD (mg/kg)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK15	HGC0165B	ND	1	NA	0.100	0.0330	03/12/1015:33	03/12/1011:00	M47C011010	M47C011008	HGC0165	NA	03/12/10
LC515	HGC0165L	0.845	1	NA	0.100	0.0330	03/12/1015:35	03/12/1011:00	M47C011011	M47C011008	HGC0165	NA	03/12/10
LC015	HGC0165C	0.848	1	NA	0.100	0.0330	03/12/1015:37	03/12/1011:00	M47C011012	M47C011008	HGC0165	NA	03/12/10
B340-0025AS	C029-06A	14.3	1	14.3	0.117	0.0385	03/12/1015:40	03/12/1011:00	M47C011013	M47C011008	HGC0165	03/02/10	03/02/10
B340-0025	C029-06	ND	1	14.3	0.117	0.0385	03/12/1015:42	03/12/1011:00	M47C011014	M47C011008	HGC0165	03/02/10	03/02/10
B340-0025DL	C029-06J	ND	5	14.3	0.583	0.193	03/12/1015:44	03/12/1011:00	M47C011015	M47C011008	HGC0165	03/02/10	03/02/10
B340-0025MS	C029-06M	1.00	1	14.3	0.117	0.0385	03/12/1015:46	03/12/1011:00	M47C011016	M47C011008	HGC0165	03/02/10	03/02/10
B340-0025MSD	C029-06S	0.996	1	14.3	0.117	0.0385	03/12/1015:49	03/12/1011:00	M47C011017	M47C011008	HGC0165	03/02/10	03/02/10
B340-0035	C029-07	ND	1	16.6	0.120	0.0396	03/12/1015:51	03/12/1011:00	M47C011018	M47C011008	HGC0165	03/02/10	03/02/10
B340-0045	C029-08	ND	1	16.1	0.119	0.0393	03/12/1015:53	03/12/1011:00	M47C011019	M47C011008	HGC0165	03/02/10	03/02/10

**Figure 4: TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

```

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG NO.: 10C029
METHOD: METHOD 7471B
=====
MATRIX: SOIL
DILTN FACTR: 1
SAMPLE ID: MBLK15
CONTROL NO.: HGC0165B
LAB FILE ID: M47C011010
DATE TIME EXTRACTD: 03/12/1011:00
DATE TIME ANALYZD: 03/12/1015:33
PREP. BATCH: HGC0165
CALIB. REF: M47C011008

% MOISTURE: NA

DATE COLLECTED: NA
DATE RECEIVED: 03/12/10

BLNK RSLT mg/kg ND
SPIKE AMT mg/kg 0.833
BS RSLT mg/kg 0.845
BS % REC 101
BSD RSLT mg/kg 0.848
BSD % REC 102
RPD % 0
QC LIMIT % 80-120
MAX RPD % 20

PARAMETER Mercury
=====
  
```

Figure 5: TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND PROJECT  
 SDG NO.: 10C029  
 METHOD: METHOD 7471B

MATRIX: SOIL  
 DILT N FACTR: 1  
 SAMPLE ID: B340-0025  
 CONTROL NO.: C029-06  
 LAB FILE ID: M47C011014  
 DATIME EXTRCTD: 03/12/1011:00  
 DATIME ANALYZD: 03/12/1015:42  
 PREP. BATCH: HGC016S  
 CALIB. REF: M47C011008

MATRIX: SOIL  
 DILT N FACTR: 1  
 SAMPLE ID: B340-0025  
 CONTROL NO.: C029-06M  
 LAB FILE ID: M47C011016  
 DATIME EXTRCTD: 03/12/1011:00  
 DATIME ANALYZD: 03/12/1015:46  
 PREP. BATCH: HGC016S  
 CALIB. REF: M47C011008

MATRIX: SOIL  
 DILT N FACTR: 1  
 SAMPLE ID: B340-0025  
 CONTROL NO.: C029-06S  
 LAB FILE ID: M47C011017  
 DATIME EXTRCTD: 03/12/1011:00  
 DATIME ANALYZD: 03/12/1015:49  
 PREP. BATCH: HGC016S  
 CALIB. REF: M47C011008

% MOISTURE: 14.3

ACCESSION:

PARAMETER	SMPL RSLT mg/kg	SPIKE AMT mg/kg	MS RSLT mg/kg	MS % REC	SPIKE AMT mg/kg	MSD RSLT mg/kg	MSD % REC	RPD %	QC LIMIT %	MAX RPD %
Mercury	ND	0.972	1.00	103	0.972	0.996	102	1	80-120	20

**Figure 6: TYPICAL DILUTION TEST REPORT SUMMARY**

EMAX QUALITY CONTROL DATA  
 SERIAL DILUTION ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND PROJECT  
 BATCH NO.: 10C029  
 METHOD: METHOD 7471B

=====

MATRIX:	SOIL		% MOISTURE:	14.3
DILUTION FACTOR:	1	5		
SAMPLE ID:	B340-0025	B340-002SDL		
EMAX SAMP ID:	C029-06	C029-06J		
LAB FILE ID:	M47C011014	M47C011015		
DATE EXTRACTED:	03/12/1011:00	03/12/1011:00	DATE COLLECTED:	03/02/10
DATE ANALYZED:	03/12/1015:42	03/12/1015:44	DATE RECEIVED:	03/02/10
PREP. BATCH:	HGC016S	HGC016S		
CALIB. REF:	M47C011008	M47C011008		

ACCESSION:

PARAMETER	SMPL RSLT (mg/kg)	SERIAL DIL RSLT (mg/kg)	DIF RSLT (%)	QC LIMIT (%)
-----	-----	-----	-----	-----
Mercury	ND	ND	0	10

**Figure 7: TYPICAL POST DIGESTION SPIKE REPORT SUMMARY**

EMAX QUALITY CONTROL DATA  
 ANALYTICAL SPIKE ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND PROJECT  
 SDG NO.: 10C029  
 METHOD: METHOD 7471B

=====

MATRIX: SOIL % MOISTURE: 14.3  
 DILTN FACTR: 1 1  
 SAMPLE ID: B340-0025  
 CONTROL NO.: C029-06 C029-06A  
 LAB FILE ID: M47C011014 M47C011013  
 DATIME EXTRACTD: 03/12/1011:00 03/12/1011:00 DATE COLLECTED: 03/02/10  
 DATIME ANALYZD: 03/12/1015:42 03/12/1015:40 DATE RECEIVED: 03/02/10  
 PREP. BATCH: HGC016S HGC016S  
 CALIB. REF: M47C011008 M47C011008

ACCESSION:

PARAMETER	SMPL RSLT (mg/kg)	SPIKE AMT (mg/kg)	AS RSLT (mg/kg)	AS % REC	QC LIMIT ( % )
Mercury	ND	0.972	1.02	105	85-115

**Figure 8:**

**TYPICAL CASE NARRATIVE**

---

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN LAND PROJECT

SDG : 10C029

METHOD 7471B  
MERCURY BY COLD VAPOR

A total of three (3) soil samples were received on 03/02/10 for Mercury analysis, Method 7471B in accordance with Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, USEPA SW-846.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source. Continuing calibration verifications were carried out at the frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG.  
Percent recoveries for HGC016SL/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at the frequency prescribed by the project.  
Percent recoveries for C029-06M/S were within project QC limits.  
Analytical spike and serial dilution were analyzed for matrix interference evaluation. Results were within method acceptance criteria.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

**Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURE**

PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> RVW	2 <sup>nd</sup> RVW
Initial multipoint calibration	Daily initial calibration prior to sample analysis	Correlation coefficient $r \geq 0.995$ for linear regression	Correct the problem then repeat initial calibration		
Initial calibration verification (second source)	Daily after initial calibration	Analyte within $\pm 10\%$ of expected value	Correct the problem then repeat initial calibration		
Calibration verification (CCV)	Daily, before sample analysis, every 10 samples and at the end of analysis sequence	Analyte within $\pm 20\%$ of expected value	Repeat calibration and re-analyze all samples since last successful calibration		
Calibration blank (CCB)	After every calibration verification	No analyte detected $> LOD$	Correct the problem then re-analyze calibration blank and previous samples		
Method blank (MB)	One per preparation batch	No analyte detected $> \frac{1}{2} LOQ$	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
Lab Control Sample (LCS)	One LCS per preparation batch	%Rec.: 80-120%	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD or MS/Dup	One set MS/MSD or MS/Dup in every preparatory batch	%Rec. 80-120%, RPD $\leq 20\%$	Perform Post-Digestion Spike.		
Post-Digestion Spike	When MS/MSD or MS/Dup fails	%Rec. 85-115%	Perform dilution test if analyte concentration is sufficiently high ( $\sim 5x$ the LOQ after dilution), otherwise perform MSA.		
Dilution Test	When Post-Digestion Spike fails and analyte concentration is sufficiently high ( $\sim 5x$ the LOQ after dilution)	Within $\pm 10\%$ of the parent sample result	Perform MSA.		
Comments: Refer to PSR for flagging criteria LOQ = lowest calibration point			Reviewed By:		
			Date:		

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

DEMONSTRATION OF CAPABILITY  
 MERCURY

SOP: EMAX-7471  
 Conc Unit: mg/Kg  
 Sample Amount(gm): 1

Extraction dates: 12/22/11 & 12/29/11  
 Analysis dates: 12/22/11 & 12/29/11  
 Extracted and Analyzed by: N. Tan

PARAMETER	HGL016SL	HGL016SC	HGL018SL	HGL018SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	M47L015-11	M47L015-12	M47L017-11	M47L017-12							
Mercury	0.887	0.875	0.900	0.910	0.833	0.893	107	0.015	1.7	80 - 120	PASSED







SOP REVIEW FORM

EMAX-8015D

Rev. 6

DIESEL RANGE ORGANICS

SOP No.

Revision Number

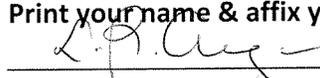
Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	✓			
Summary of Method	✓			
Detection Limits	✓			
Dynamic Range	✓			
Sample Holding Time & Preservation	✓			
Associated SOPs	✓			
Safety	✓			I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	✓			
Standards	✓			
Procedures	✓			
- Sample Preparation	✓			
- Instrument Parameters	✓			
- Calibration	✓			
- Analysis	✓			
- Calculations	✓			
- Data Reduction	✓			
- Report Generation	✓			
- Data Review	✓			
- Preventive Maintenance	✓			
Quality Control	✓			
Corrective Action	✓			
Pollution Prevention	✓			
Waste Management	✓			
Supplementary Notes	✓			
References	✓			
Appendices	✓			

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

  
LUCITA R. ARZADON

Date: 09/24/14

## STANDARD OPERATING PROCEDURES

**DIESEL RANGE ORGANICS**

SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-13

Prepared By: Lucita Arzadon *L.A. Arzadon* Date: 09/30/13

Approved By: Kenette Pimentel *K. Pimentel* Date: 09-30-13  
QA Manager

Approved By: Caspar Pang *C. Pang* Date: 09-30-13  
Laboratory Director

Control Number: **8015D-06-**

**1.0 SCOPE AND APPLICATION**

- 1.1 This method is used to analyze extractable fuel hydrocarbons in water, soil and other sediment samples. In the vast field of petroleum hydrocarbons, this method is limited to provide semi-quantitative results on those extractable hydrocarbons with a comparable aliphatic hydrocarbon range from C<sub>10</sub> to C<sub>34</sub>.
- 1.2 This SOP is an adaptation of SW846 Method 8015C. This SOP is also applicable to SW846 Method 8015B and Method 8015D.

**2.0 SUMMARY OF METHOD**

- 2.1 Petroleum hydrocarbons are extracted in methylene chloride, analyzed by flame ionization detector (FID) in gas chromatograph and quantified as diesel fuel at C<sub>10</sub> to C<sub>28</sub> range. The hydrocarbons that fall in this range are defined as Diesel Range Organics (DRO).
- 2.2 Other fuel standards that fall in the range of C<sub>10</sub> to C<sub>34</sub> can also be quantitated by this method.
- 2.3 **Interference**
- 2.3.1 Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to its use.
- 2.3.2 Carry-over from a highly concentrated sample can be potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.
- 2.3.3 Deposits may adhere in the injection port/glass liner over a period of time and can cause interference. The injection port and glass liner must be routinely cleaned.

**3.0 DETECTION LIMITS**

- 3.1 **Detection Limit (DL), Limit of Detection (LOD) and Limit of Quantitation (LOQ)**
- 3.1.1 Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2 Refer to Table 1 for established limits.

**4.0 DYNAMIC RANGE**

## STANDARD OPERATING PROCEDURES

**DIESEL RANGE ORGANICS**SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-13

- 4.1 The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (refer to Section 9.5.1). Dilute and re-analyze all samples having results above this range to properly quantitate.
- 4.2 The lowest quantifiable range of diluted samples is equal to the lowest calibration point (refer to Section 9.5.1). Lower the dilution factor and re-analyze all diluted samples analyzed below this range to properly quantitate.

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1 Sample Collection**

- 5.1.1 Water samples received in the lab are expected to be contained in an amber bottle with Teflon-lined cap and cooled to  $\leq 6^{\circ}\text{C}$  without freezing.
- 5.1.2 Soil samples received in the lab are expected to be contained in a jar or Shelby tube and cooled to  $\leq 6^{\circ}\text{C}$  without freezing.

**5.2 Holding Time**

- 5.2.1 Water samples must be extracted within 7 days from sampling date.
- 5.2.2 Soil samples must be extracted within 14 days from sampling date.
- 5.2.3 All extracts must be analyzed within 40 days from extraction.

**5.3 Preservation**

- 5.3.1 Store water samples at  $\leq 6^{\circ}\text{C}$  without freezing, store in dark.
- 5.3.2 Store soil samples and extracts at  $\leq 6^{\circ}\text{C}$  without freezing.

**6.0 ASSOCIATED SOPs**

- 6.1 EMAX-3520 Extraction, Continuous Liquid/Liquid
- 6.2 EMAX-3540 Extraction, Soxhlet
- 6.3 EMAX-3550 Extraction, Pulse Sonication
- 6.4 EMAX-3580 Waste Dilution
- 6.5 EMAX-DM01 Data Flow & Review
- 6.6 EMAX-QA04 Detection Limit (DL)
- 6.7 EMAX-QA05 Training
- 6.8 EMAX-QA08 Corrective Action
- 6.9 EMAX-QC01 Quality Control for Chemicals
- 6.10 EMAX-QC02 Analytical Standard Preparation
- 6.11 EMAX-QC07 Glassware Cleaning
- 6.12 EMAX-SM01 Sample Management

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- 6.13 EMAX-SM03 Waste Disposal  
 6.14 EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1 Read all SDS for chemicals listed in this SOP.  
 7.2 Treat reagents, standards and samples as potential hazard. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.  
 7.3 If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENT, CHEMICALS & REAGENTS****8.1 Instruments and Supplies**

## 8.1.1 Gas Chromatography

Instrument ID	Inst. D5	Inst. F2
Gas Chromatography and Autosampler	Agilent 6890 with 7683B autosampler	Perkin Elmer Clarus 680
Detector	FID	FID
Column	HP-5 30m x 0.32 x 0.25 $\mu$ m	HP-5 30m x 0.32 x 0.25 $\mu$ m
Data Acquisition	EZChrom Elite 3.3.1	EZChrom Elite 3.3.2

8.1.2 Syringes: 10, 25, 100  $\mu$ l microsyringe

8.1.3 Volumetric Flask: 10, 100 and 1000 ml

8.1.4 Vials: 2, 10 and 40 ml, amber

8.1.5 Bottle: 250 ml (amber)

**8.2 Chemicals and Reagents**

- 8.2.1 Where available, purchase reagent-grade chemicals and reagents.  
 8.2.2 Methylene Chloride  
 8.2.3 Acetone, Methanol  
 8.2.4 High purity He, H<sub>2</sub>, Air, N<sub>2</sub>

**9.0 STANDARDS****9.1 Standard Preparation**

- 9.1.1 Refer to EMAX-QC02 for proper analytical standard preparation.

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9.1.2 Other concentration levels may be prepared as long as it complies with the method and project requirements.

9.2 **Stock Standard**9.2.1 Diesel

Purchase stock standards as certified solutions from certified vendors as listed below or equivalent:

Standard Name	Source	Concentration (mg/L)	Solvent	Intended Use
Diesel	Restek	50,000	MeCl <sub>2</sub>	ICAL, DCC
Diesel	CPI	50,000	Acetone	ICV, LCS/LCSD, MS/MSD
Bromobenzene / Hexacosane	CPI	2000 / 500	Acetone/MeCl <sub>2</sub> 1:1	Surrogates

9.2.2 JP5 and Motor Oil

Purchase stock standards as certified solutions from certified vendors as listed below or equivalent:

Standard Name	Source	Concentration (mg/L)	Solvent	Intended Use
JP-5 Military Fuel	Supelco	10,000	MeCl <sub>2</sub>	ICAL, DCC
JP-5	AccuStandard	20,000	Methanol	ICV, LCS/LCSD, MS/MSD
Motor Oil 5W30	Restek	50,000	MeCl <sub>2</sub>	ICAL, DCC
SAE 5W30 Motor Oil	AccuStandard	Neat	NA	ICV, LCS/LCSD, MS/MSD

9.3 **Intermediate Standard**

9.3.1 Prepare SAE 5W30 Motor Oil Standard with Methylene Chloride as follows:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
SAE 5W30 Motor Oil	Neat	1.0 g	100,000 mg/L	MeCl <sub>2</sub>	10 ml

9.3.2 Prepare with water-soluble solvent Acetone as follows:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
Intermediate Standard	100,000 mg/L	5.0 ml	5,000 mg/L	Acetone	100 ml

9.4 **Calibration Standard**9.4.1 Initial Calibration Standard9.4.1.1 **Diesel**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in MeCl<sub>2</sub> and store in Teflon-sealed vial with minimal headspace; suggested concentration and injection volume are as follows:

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ICAL Pt.	Stock Standard (50,000 mg/L) Aliquot ( $\mu$ l)	Final Volume (ml)	Final Concentration (mg/L)
1	0.1	1	5
2	0.2	1	10
3	1	1	50
4	2	1	100
5	10	1	500
6	30	1	1500
7	60	1	3000

9.4.1.2 **JP5**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in MeCl<sub>2</sub> and store in Teflon-sealed vial with minimal headspace

ICAL Pt.	Stock Standard (10,000 mg/L) Aliquot ( $\mu$ l)	Final Volume (ml)	Final Concentration (mg/L)
1	1	1	10
2	5	1	50
3	10	1	100
4	50	1	500
5	150	1	1500
6	300	1	3000

9.4.1.3 **Motor Oil**

Prepare a minimum of 5-point calibration standard (ideally 6-point) from the primary stock standard in MeCl<sub>2</sub> and store in Teflon-sealed vial with minimal headspace

ICAL Pt.	Stock Standard (50,000 mg/L) Aliquot ( $\mu$ l)	Final Volume (ml)	Final Concentration (mg/L)
1	0.2	1	10
2	1	1	50
3	2	1	100
4	10	1	500
5	30	1	1500
6	60	1	3000

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**DIESEL RANGE ORGANICS**SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-139.4.2 Initial Calibration Verification Standard9.4.2.1 **Diesel**

Prepare initial calibration verifications standard (using secondary source standard) in MeCl<sub>2</sub>.

<b>Diesel Std. (50,000 mg/L) Aliquot (μl)</b>	<b>Bromobenzene / Hexacosane (2000 / 500 mg/L) Aliquot (μl)</b>	<b>Final Volume (ml)</b>	<b>Final Conc. Analyte/Surrogates (mg/L)</b>
10	40	1	500 / 80/ 20

9.4.2.2 **JP5/Motor Oil**

Prepare initial calibration verifications standard (using secondary source standard) in MeCl<sub>2</sub>.

<b>JP5 Std. (20,000 mg/L) Aliquot (μl)</b>	<b>Motor Oil Std. (Intermediate Std.) (5,000 mg/L) Aliquot (μl)</b>	<b>Final Volume (ml)</b>	<b>Final Conc. JP5/Motor Oil (mg/L)</b>
25	100	1	500 / 500

9.4.3 Continuing Calibration Standard9.4.3.1 **Diesel**

Prepare daily calibration standards (using primary-source standards) in MeCl<sub>2</sub>.

<b>Diesel Std. (50,000 mg/L) Aliquot (μl)</b>	<b>(2000 / 500 mg/L) Aliquot (μl)</b>	<b>Final Volume (ml)</b>	<b>Final Conc. Diesel/Surrogates (mg/L)</b>
100	400	10	500 / 80/20

9.4.3.2 **JP5/Motor Oil**

Prepare daily calibration standards (using primary-source standards) in MeCl<sub>2</sub>.

<b>JP5 Std. (10,000 mg/L) Aliquot (μl)</b>	<b>Motor Oil Std. (50,000 mg/L) Aliquot (μl)</b>	<b>Final Volume (ml)</b>	<b>Final Conc. JP5/Motor Oil/Surrogates (mg/L)</b>
500	100	10	500 / 500

9.4.4 Surrogate Initial Calibration Standard

9.4.4.1 Prepare a minimum of 5-point calibration standard (ideally 6-point) from the surrogate standard in MeCl<sub>2</sub> and store in Teflon sealed vial with minimal headspace; suggested concentration and injection volume are as follows:

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ICAL Pt.	Surrogate Standard (2000/500 mg/L) Aliquot (µl)	Final Volume (ml)	Final Concentration – Bromobenzene / Hexacosane (mg/L)
1	10	1	20 / 5
2	20	1	40 / 10
3	30	1	60 / 15
4	40	1	80 / 20
5	50	1	100 / 25
6	110	1	220 / 55

**9.5 LCS/MS Spike Standard**

9.5.1 Use secondary source standards or primary source standards for laboratory control standard (LCS) and matrix spike (MS) standards.

**9.6 Retention Time Window Standard**

9.6.1 Purchase the following standards or equivalent.

Standard Name	Source	Concentration	Solvent
Calibration Window Defining Hydrocarbon (C <sub>8</sub> – C <sub>40</sub> )	AccuStandard	1000	Chloroform
Hydrocarbon Window Defining Standard (C <sub>9</sub> – C <sub>39</sub> )	AccuStandard	500	Hexane

9.6.2 Prepare these n-alkane standards with MeCl<sub>2</sub> as follows:

Standard Name	Concentration	Amount	Final Volume	Final Concentration
C <sub>8</sub> -C <sub>40</sub> RTW Std.	1000 mg/L	0.2 ml	10 ml	20 mg/L
C <sub>9</sub> -C <sub>39</sub> RTW Std.	500 mg/L	0.4 ml		20 mg/L

**10.0 PROCEDURES****10.1 Sample Preparation****10.1.1 Aqueous Samples**

10.1.1.1 Prepare aqueous samples in accordance with EMAX-3520, unless otherwise specified by the project.

**10.1.2 Soil Samples**

10.1.2.1 Prepare soil samples in accordance with any of the following extraction procedures: EMAX-3540, EMAX-3550 or EMAX-3580 as specified by the project. Where project does not specify extraction procedure, default to EMAX-3550.

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**DIESEL RANGE ORGANICS**SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-13**10.2 Instrument Parameters**

10.2.1 Fine tune the instrument guided by parameter conditions as listed below:

Injector Temperature – 280°C

Detector Temperature – 320°C

Head Pressure – 21 psi

Temperature Program

Instrument	D5	F2
Initial Temperature	55°C, hold for 0.5 min	60°C, hold for 0.5 min
Temperature 1	320°C, hold for 5 min	310°C, hold for 4 min
Rate 1	60°C /min	55°C /min
Temperature 2	-	320°C, hold for 2 min
Rate 2	-	60°C /min
Total Run Time	10 min	12 min

**10.3 Calibration****10.3.1 Initial Calibration**

10.3.1.1 Analyze the initial calibration standards prepared. Sum the area of all peaks eluting between C<sub>10</sub> to C<sub>28</sub> for each of the calibration point. Generate this area by rejoining a horizontal baseline between the retention time of C<sub>10</sub> to C<sub>28</sub>. Check that the highest point does not have saturated peak(s).

*Note: The lowest calibration determines the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.*

**10.3.2 Initial Calibration Verification (ICV)**

10.3.2.1 After establishing ICAL, analyze the ICV standard (Refer to Section 9.4.2) to verify the validity of the ICAL. Refer to Appendix 1 for acceptance criteria. If non-compliant, refer to Section 12 for corrective action.

**10.3.3 Retention Time Window Check (RTW)**

10.3.3.1 Analyze the RTW standard after every ICAL to set carbon cut-off ranges (e.g., C<sub>10</sub>-C<sub>24</sub>, C<sub>10</sub>-C<sub>28</sub>). Refer to Section 9.6.

**10.3.4 Daily Continuing Calibration (DCC)**

10.3.4.1 Analyze a continuing calibration (Refer to 9.4.3) at the beginning of a 12-hour shift or as specified by the project.

10.3.4.2 Refer to Appendix 1 for acceptance criteria and corrective action.

**10.4 Analysis****10.4.1 Extract Preparation**

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10.4.1.1 Allow the extracts to equilibrate to room temperature.

10.4.1.2 Transfer about 1 ml of extracts into autosampler vials.

10.4.2 **Analytical Sequence**

10.4.2.1 Analyze instrument blank to ensure that the instrument is free from contamination.

10.4.2.2 Analyze DCC to check ICAL validity.

10.4.2.3 Analyze Method Blank to check for preparation batch contamination.

10.4.2.4 Analyze Lab Control Sample to check accuracy.

10.4.2.5 Analyze Lab Control Sample Duplicate (if required by the project).

10.4.2.6 Analyze samples to a maximum of 12-hour runs or as specified by the project.

10.4.2.7 Analyze matrix spikes (MS/MSD) per project requirement.

10.4.2.8 Record the analytical sequence in the Analytical Run Log.

10.4.2.9 Print instrument sequence before and after the analysis run and attach to the analytical run. Document any changes that occurred during the process.

10.4.3 **Identification and Quantitation**

10.4.3.1 Identification is based on pattern recognition. Hence, compare sample chromatograms to reference hydrocarbons standard chromatograms for their response hydrocarbon range and peak distribution to determine the most probable petroleum product.

10.4.3.2 All peaks eluting within the established RT window identifies the DRO, JP5 and Motor Oil.

10.4.3.3 When the elution profile of a sample does not match that of diesel standard, JP5 or motor oil, but falls within the retention time window, quantitate results as diesel range organics (DRO) and denote the observed deviation in case narrative.

10.4.3.4 Quantitation is achieved by the summation of all peaks in the chromatogram minus the solvent peak, and the sample result is calculated using Eq.-10.5.3.

10.4.3.5 Integrate the total peak area response and quantitate the total area by using the ACF of Diesel, JP5 or Motor Oil (refer to Eq.-10.5.1.2).

10.4.3.6 When manual integration is necessary follow the procedures described in EMAX-DM01 (Section for manual integration).

10.4.4 **Retention Time Window (RTW)**

10.4.4.1 **Establishing RTW**

10.4.4.1.1 Run RTW standard over a period of 72 hours.

10.4.4.1.2 Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte (use Eq.-10.5.1.3).

10.4.4.1.3 The width of RTW is defined by  $\pm 3XSD$ .

10.4.4.2 **Evaluating RTW**

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10.4.4.2.1 If the SD is equal to 0.00, default to the previous study until historical data is obtained to define the RTW for the current instrument.

10.4.4.2.2 For new instruments, use the established retention time from another instrument having the same instrument parameters (e.g. detector, temperature program and column). If there are no instruments with the same instrument parameter, use 0.03 minutes as the default RTW until historical data is obtained to define the RTW for the current instrument parameters condition.

**10.4.4.3 Application of RTW**

10.4.4.3.1 Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.4.3.2 Whenever the observed retention is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

**10.4.4.4 Updating RTW**

10.4.4.4.1 Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur.

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of the RTW)
- Major instrument maintenance (e.g. replacement of detector).

10.4.4.4.2 If the calculated new RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

**10.4.5 Sample Result Evaluation**

10.4.5.1 Check QC Criteria as soon as available.

- Check LCS, MS surrogate recoveries against Project Specific Requirement (PSR). In the absence of PSR, default to in-house QC limits. Refer to Appendix 1.

**10.4.5.2 Qualitative Identification**

- Compare the sample chromatogram to the pattern established by the calibration standard.
- When peaks other than diesel pattern are detected within the DRO, take note of it and discuss in the case narrative.

**10.4.5.3 Quantitation**

- Assign the appropriate hydrocarbon range for quantitation (e.g. C<sub>10</sub> – C<sub>28</sub>; C<sub>18</sub> – C<sub>34</sub>; C<sub>8</sub>-C<sub>18</sub>; etc.).

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- Integrate all peaks bracketed by the hydrocarbon range baseline to the baseline. Set the integration range at least 95% within of the total spectrum of the expected DRO range.
- Calculate the result as specified in Figure 2 based on the appropriate fuel standard (e.g. Diesel: C<sub>10</sub> – C<sub>28</sub>; Motor Oil: C<sub>18</sub> – C<sub>34</sub>; JP5 C<sub>8</sub>-C<sub>18</sub>). In the absence of alkane range specification for the project, request for directive from the PM.
- Check that all positively identified target analytes are quantitated within the calibration range.
- Dilute and re-analyze all positively identified target analytes exceeding calibration range.
- When peaks other than diesel pattern are detected within the DRO range, quantitate using the DRO calibration factor and discuss it in the case narrative (e.g. peaks within the DRO range does not resemble diesel pattern or motor oil pattern). When saturated peaks are present, dilute the extract appropriately until peaks are eluted properly.

**10.5 Calculations****10.5.1 Initial Calibration****10.5.1.1 Calculate for Calibration Factor (CF)**

$$CF = \frac{R_t}{C_v} \quad \text{Eq.10.5.1.1}$$

where:

- $R_t$  – Total response of the integrated peaks  
 $C_v$  – Known value of the standard concentration, mg/L

**10.5.1.2 Calculate for the Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF}{n} \quad \text{Eq.-10.5.1.2}$$

where:

- $\sum CF$  – Summation of Calibration Factors  
 $n$  – Number of measurements

**10.5.1.3 Calculate for Standard Deviation (SD)**

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq.-10.5.1.3}$$

where:

- $X_i$  – Result at the  $i^{\text{th}}$  measurement

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**DIESEL RANGE ORGANICS**SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-13 $\bar{x}$  – Mean $n$  – Number of measurements**10.5.1.4 Calculate for Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{ACF} \times 100 \quad \text{Eq.-10.5.1.4}$$

where:

 $SD$  – Standard deviation $ACF$  – Average Calibration Factor**10.5.2 Calculate the Percent Difference of DCC from ACF**

$$\%D = \frac{C_f - C_k}{C_k} * 100 \quad \text{Eq.-10.5.2}$$

where:

 $\%D$  – Percent Difference DCC from known concentration $C_k$  – Known concentration of the analyte, in mg/L $C_f$  – Found concentration, in mg/L**10.5.3 Calculate for Sample Concentration****10.5.3.1 Water Samples**

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)} \quad \text{Eq.-10.5.3.1}$$

where:

 $C$  – Concentration of the sample, mg/L $R_t$  – Total response of the integrated peaks $V_e$  – Volume of Extract, ml $A_s$  – Sample amount, ml $DF$  – Dilution Factor $ACF$  – Average Calibration Factor**10.5.3.2 Soil Samples**

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)(\%S)} \quad \text{Eq.-10.5.3.2}$$

where:

 $C$  – Concentration of the sample, mg/Kg

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- $R_t$  – Total response of the integrated peaks  
 $V_e$  – Volume of Extract, ml  
 $A_s$  – Sample amount, g  
 $DF$  – Dilution Factor  
 $ACF$  – Average Calibration Factor  
 $\%S$  – Percent solids (%S of water = 1)

10.5.4 **Percent Recovery**

$$\% \text{ Recovery} = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.5.4}$$

where:

- $C_f$  – Concentration found  
 $C$  – Concentration of the sample (use 0 for LCS)  
 $C_s$  – Concentration of spike

10.5.5 **Relative Percent Difference (RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.5}$$

where:

- $C_1$  – Measured concentration of the first sample aliquot  
 $C_2$  – Measured concentration of the second sample aliquot

10.6 **Data Reduction**

- 10.6.1 Make a copy of the analytical run log.  
 10.6.2 Print a copy of the raw data and the QC report.  
 10.6.3 Highlight the data to be reported.  
 10.6.4 Collate the reportable data separating the QC results from the sample results.  
 10.6.5 Keep all other data generated with the analytical folder marked with "For record only".

10.7 **Report Generation**

- 10.7.1 Generate the method.txt file using WDBX<sup>1</sup>CN.exe.  
 10.7.2 Generate the sample results using F1NVX<sup>1</sup>C.exe or F1NVX<sup>1</sup>C4.exe.  
 10.7.3 Generate the QC summary using QCVX<sup>1</sup>CN.exe or QCVX<sup>1</sup>CN4.exe.

<sup>1</sup> X – version number

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10.7.4 Generate the Lab Chronicle using LABCHRN1.exe.

10.7.5 Generate the Case Narrative using CN1.exe.

**10.8 Data Review**

10.8.1 Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analysis.

10.8.1.1 Case Narrative

10.8.1.2 Lab Chronicle

10.8.1.3 Sample Results

10.8.1.4 Method Blank Results

10.8.1.5 LCS/LCSD Summary

10.8.1.6 MS/MSD Summary

10.8.1.7 ICAL Summary

10.8.1.8 ICV Summary

10.8.1.9 DCC Summary

10.8.1.10 Analytical Run Log

10.8.1.11 Sample Preparation Log

10.8.1.12 Non-Conformance Report (if any)

10.8.2 Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.2.1 If any of the checkpoints below indicate a problem, re-analysis is required.

- ✓ Check that qualitative identification is done properly.
- ✓ Check surrogate recoveries against Project Specific Requirements (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check that all samples results are integrated properly and results over calibration range are diluted and re-analyzed within the calibration range.
- ✓ Where manual integration was performed, check that it was done properly and documentation was retained in accordance to EMAX-DM01 (Section for manual integration).
- ✓ Check that saturated peak(s) are diluted and quantitated properly.
- ✓ Check that suspected carry-overs are re-analyzed and results are reported accordingly.
- ✓ Check that discrete peaks (other than column bleeds) are reported according to project requirement.

10.8.2.2 Review the attached logs that they are properly filled.

10.8.2.3 Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

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10.8.2.4 Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3 Submit the analytical folder for secondary review.

### 10.9 Preventive Maintenance

10.9.1 Perform daily routine check and record it in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control. Refer to Form 8015DFM for daily routine maintenance check points. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2 The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

<b>Maintenance Activity</b>	<b>Description</b>	<b>Frequency</b>
Autosampler	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Change liner as necessary. Check instrument performance (e.g., Daily calibration check, instrument blank).	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector. Check septa and replace as necessary.	Every 6 months or as necessary
Complete Inspection	Perform general inspection of the complete system Inspect autosampler cabling and configuration setting. Inspect column, change if necessary (6 mo. or as needed)	Once a year

10.9.3 Maintain an inventory of instrument parts and supplies for routine maintenance.

## 11.0 **QUALITY CONTROL**

### 11.1 Analytical Batch QC

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- 11.1.1 Initial Calibration must be established and verified by daily continuing calibration as described in Appendix 1.
- 11.1.2 Analytical batch must consist of a valid ICAL, QC Samples and field samples bracketed with opening and closing DCC every 12-hour analytical sequence, unless other frequency is prescribed by the project.
- 11.1.3 A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing a solvent blank and identifying its result as instrument blank.
- 11.2 **Preparation Batch QC**
- 11.2.1 A preparation batch consists of a MB, LCS, MS/MSD and  $\leq 20$  field samples.
- 11.2.2 For water samples, use organic-free water for MB and LCS.
- 11.2.3 For soil samples, use organic-free sand for MB and LCS.
- 11.2.4 Prepare, analyze and control QC samples as required by the project. In the absence of PSR, refer to Appendix 1 for Quality Control Procedures.
- 11.2.5 Surrogate standard must be added to all samples, including quality control samples (e.g., method blank, LCS and MS). Check the PSR for QC control Limits. In the absence of PSR default to EMAX-QC Limits.
- 11.2.6 Solvents and reagents must undergo quality control check prior to use. Refer to EMAX-QC01 for details.
- 11.2.7 Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.
- 11.3 **Method QC**
- 11.3.1 All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.
- 11.3.2 Instrument performance must be checked prior to analysis.
- 11.3.3 Retention Time Window must be established and updated as prescribed.
- 11.3.4 All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.
- 11.4 Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

**12.0 CORRECTIVE ACTION**

- 12.1 Corrective action for each Quality Control procedure is summarized in Appendix 1.
- 12.2 **Calibration**
- 12.2.1 If initial calibration is non-compliant, consider the following suggestions:
- 12.2.1.1 If RSD > 20%, check each calibration point. If an outlier exists, re-analyze that calibration point.
- 12.2.1.2 If ICV is not within the expected recovery range, review the chromatogram.
- Bias low results are indicative of bad injection or standard degradation.

## STANDARD OPERATING PROCEDURES

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- Bias high is indicative of inaccurate standard injection of instrument or contamination.
- Consider preparing a fresh ICV standard and re-analyze the ICV.

12.2.1.3 If problem persists, inform the Supervisor.

12.2.2 If the continuing calibration is non-compliant, consider the suggestions described in correcting the ICV. Consider also the following suggestions to correct the problem:

- Change the liner
- Clean the injection port
- Cut or replace the column
- Clean the detector
- Rule-out leaks by checking all connections
- If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL

12.2.3 If the instrument blank is non-compliant, consider the following suggestions to correct the problem:

12.2.3.1 Check the solvent blank source, e.g. check if same source was used by a similar analysis on a different instrument to rule out solvent contamination.

12.2.3.2 Bake the GC column for at least 15 min.

12.2.3.3 Re-calculate the data and/or re-analyze the extract, if any of the above checks reveal a problem.

12.2.3.4 If problem persists, inform the Supervisor prior to re-analysis.

### 12.3 **Surrogates**

12.3.1 If surrogates are non-compliant, and are not due to matrix effects, consider the following suggestions to correct the problem:

12.3.1.1 Check that the surrogate peak is properly integrated.

12.3.1.2 Check for calculation errors and that the concentrations of the surrogate solutions are correct.

- High recoveries may be due to co-eluting matrix interference, examine the sample chromatogram.
- Low recoveries may be due to bad injection during preparation process and/or analytical process.

12.3.1.3 Check instrument performance to determine if it is within acceptable guidelines.

### 12.4 **Preparation Batch QC**

12.4.1 For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.4.2 if method blank is non-compliant, consider the following suggestion to possibly correct the problem:

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- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM.
- If sample results are not reportable, determine the source of contamination and correct the problem. Re-analyze method blank and all samples processed with the contaminated blank.

12.4.3 If LCS is non-compliant, consider the following suggestions to possibly correct the problem:

- Check the results, if standard degradation is apparent, prepare a fresh standard and perform the corrective action as described in Appendix 1.
- If the LCS result is bias high, the solvent of the standard may have evaporated. Prepare a fresh standard and perform the corrective action as described in Appendix 1.
- Check instrument performance to determine if it is within acceptable guidelines.
- Re-analyze the extract if any of the above checks reveal a problem.
- Otherwise, re-extract all samples associated with the non-compliant LCS with a new set of QC samples.

12.5 A Non-Conformance Report (NCR) is required when any of the following circumstances occur.

- Anomalies, other than specified in Appendix 1, are observed.
- Sample is out of technical holding time.

12.5.1 Refer to EMAX-QA08 for NCR details.

**13.0 POLLUTION PREVENTION**

13.1 Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2 Prepare all standards in fume hoods.

**14.0 WASTE MANAGEMENT**

14.1 No samples may be dumped on the laboratory sink.

14.2 Separate and properly identify all unused expired analytical standards for proper disposal.

14.3 Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4 Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1 Definition of Terms**

15.1.1 Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belongs to the same chemical family, and which are analyzed together.

## STANDARD OPERATING PROCEDURES

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- 15.1.2 **Batch** – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.2.1 **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.2.2 **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.3 **Detection Limit (DL)** – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.1.4 **Limit of Detection (LOD)** – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.
- 15.1.5 **Limit of Quantitation (LOQ)** – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.1.6 **Safety Data Sheet (SDS)** – is a written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.1.7 **Calibration** – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8 **Calibration Blank** – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.9 **Instrument Method** – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.10 **Method Blank** – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.11 **Lab Control Sample (LCS)** – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.12 **Lab Control Sample Duplicate (LCSD)** – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.13 **Sample** – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.14 **Sample Duplicate** – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

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15.1.15 Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.16 Matrix – is a component or form of a sample.

15.1.17 Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.18 Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.19 Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.20 Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

**15.2 Application of EMAX QC Procedures**

15.2.1 The procedures and QC criteria summarized in this SOP applies to all projects when performing Diesel Range Organics Analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

**15.3 Department of Defense (DoD) Projects**

15.3.1 Samples from DoD sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

**15.4 Department of Energy (DoE) Projects**

15.4.1 Samples from DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, is applied.

**16.0 REFERENCES**

- 16.1 US EPA Method 8015C, Revision 3, February 2007
- 16.2 US EPA Method 8015D, Revision 4, June 2003
- 16.3 US EPA Method 8015B, Revision 2, December 1996
- 16.4 US EPA Method 8000B, Revision 2, December 1996
- 16.5 EMAX Quality Systems Manual, as updated

**17.0 APPENDICES****17.1 Tables**

17.1.1 Table 1 Established DL, LOD and LOQ

**17.2 Figures**

## STANDARD OPERATING PROCEDURES

**DIESEL RANGE ORGANICS**SOP No.: EMAX-8015D Revision No. 6 Effective Date: 30-Sep-13

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- 17.2.1 Figure 1 Peak Evaluation Technique
  - 17.2.2 Figure 2A Typical DRO Chromatogram
  - 17.2.3 Figure 2B Typical JP5 & Motor Oil Chromatogram
  - 17.2.4 Figure 2C Typical n-Alkane Chromatogram
  - 17.2.5 Figure 3A Typical Diesel Initial Calibration Summary
  - 17.2.6 Figure 3B Typical JP5 & Motor Oil Initial Calibration Summary
  - 17.2.7 Figure 4A Typical Diesel Continuing Calibration Summary
  - 17.2.8 Figure 4B Typical JP5 & Motor Oil Continuing Calibration Summary
  - 17.2.9 Figure 5 Typical Raw Data
  - 17.2.10 Figure 6 Typical Sample Result Summary
  - 17.2.11 Figure 7 Typical LCS/LCSD Summary
  - 17.2.12 Figure 8 Typical MS/MSD Summary
  - 17.2.13 Figure 9 Typical Case Narrative
  - 17.3 **Appendices**
    - 17.3.1 Appendix 1 Summary of Quality Control Procedures
    - 17.3.2 Appendix 2 Demonstration of Capability
  - 17.4 **Forms**
    - 17.4.1 8015DFS Sample Preparation Log
    - 17.4.2 8015DFA Analytical Run Log
    - 17.4.3 8015DFM Instrument Maintenance Log

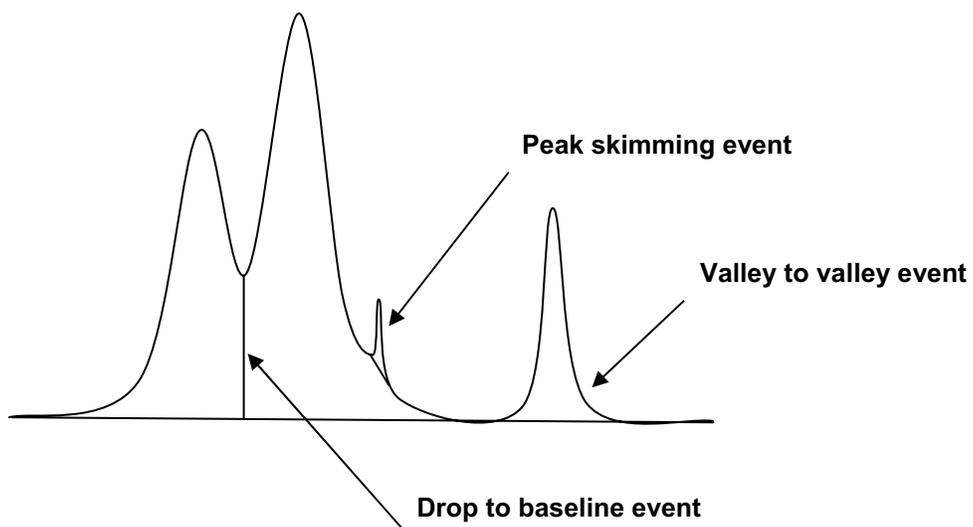
**Table 1: ESTABLISHED DL, LOD AND LOQ**

Parameter	Water (mg/L)			Soil (mg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
Diesel (Total)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C24)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C25)	0.025	0.05	0.1	2.5	5	10
Diesel (C10-C28)	0.025	0.05	0.1	2.5	5	10
JP5	0.025	0.05	0.1	2.8	5	10
5W30	0.025	0.05	0.1	2.5	5	10
10W30	0.025	0.05	0.1	-	-	-
Bromobenzene	0.05	0.1	0.2	5.0	10	20
Hexacosane	0.0125	0.025	0.05	1.25	2.5	5

Figure 1:

PEAK EVALUATION TECHNIQUE

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**Figure 2A: TYPICAL DRO CHROMATOGRAM**

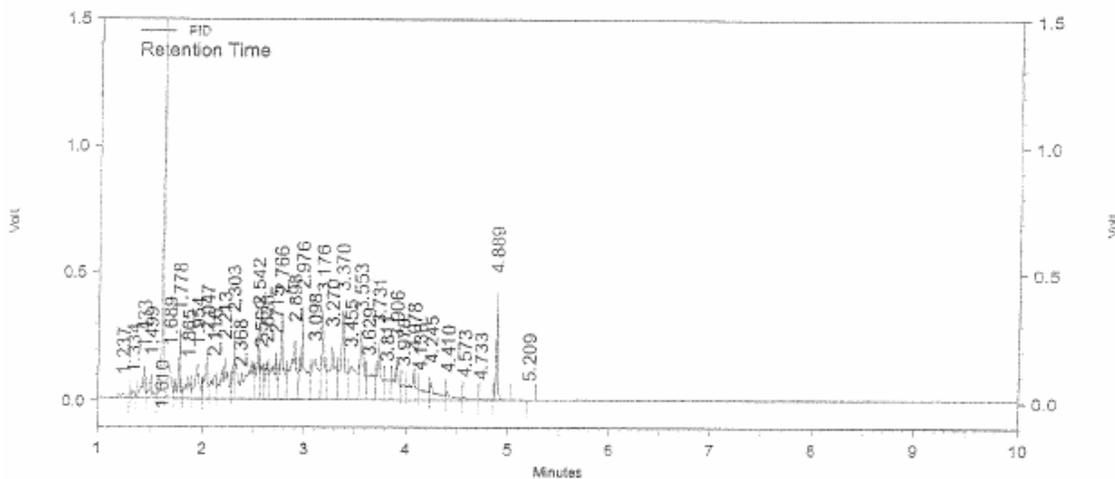
METHOD 8015 by GC/FID  
 EMAX Laboratories, Inc.

Inst. Name: : GCT-105 (Offline)  
 File : D:\Projects\EZC331\Data\LE24\LE24010.dat  
 Method : D:\Projects\EZC331\Method\DSD5E24.met  
 Sequence: : D:\Projects\EZC331\Sequence\LE25.seq  
 Sample ID : IDSD5E2401 DSL 500/80/20PPM  
 Acquired : 05/24/13 13:44:00  
 Printed : 05/24/13 17:21:28  
 User : CHERRY

**FID Results**

Name	Retention Time	Area	Average RF	ESTD conc. [ ppm ]
BROMOBENZENE	1.610	1514416 ✓	19037.34242	79.550 ✓
HEXACOSANE	4.889	498802 ✓	24896.70051	20.035 ✓
DIESEL(TOTAL)		16393833 ✓	31462.71743	521.056 ✓
DIESEL(C10-C24)		15436018 ✓	30401.78600	507.734 ✓
DIESEL(C10-C28)		15502854 ✓	30544.07919	507.557 ✓
DIESEL(C10-C25)		15486749 ✓	30519.36600	507.440 ✓
DIESEL(C9-C24)		15944064 ✓	30968.89186	514.841 ✓
DIESEL(C9-C25)		15994795 ✓	31086.47186	514.526 ✓
DIESEL(C10-C36)		15503938 ✓	30544.88929	507.579 ✓
DIESEL(C10-C40)		15503938 ✓	30544.88929	507.579 ✓

Totals		127779407		4187.896
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AK  
 05/28/13

Software Version: Version 3.3.1

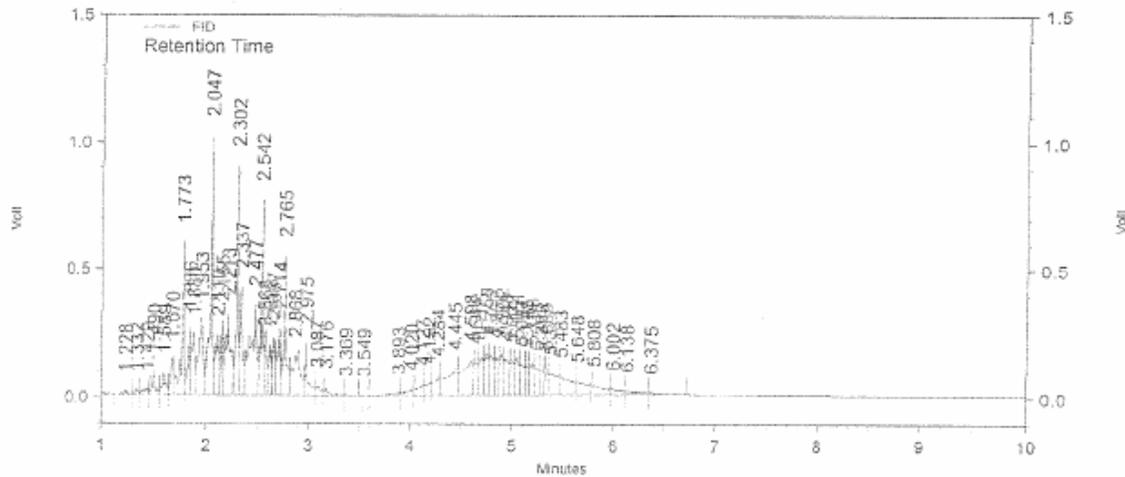
**Figure 2B: TYPICAL JP5 & MOTOR OIL CHROMATOGRAM**

METHOD 8015 by GC/FID  
 EMAX Laboratories, Inc.

Inst. Name: : GCT-105 (Offline)  
 File : D:\Projects\EZC331\Data\LE24\LE24017.dat  
 Method : D:\Projects\EZC331\Method\DSD5E24.met  
 Sequence: : D:\Projects\EZC331\Sequence\LE25.seq  
 Sample ID : IDSD5E2402 JP5/5W30 500/500PPM  
 Acquired : 05/24/13 16:16:11  
 Printed : 05/24/13 17:30:23  
 User : CHERRY

FID Results

Name	Retention Time	Area	Average RF	ESTD conc. [ ppm ]
JP5(C8-C18)		15505176 ✓	31608.84361 ✓	490.533 ✓
M.OIL(C18-C36)		10072865 ✓	19563.21878 ✓	514.888 ✓
M.OIL(C24-C36)		8426268 ✓	16606.39789 ✓	507.411 ✓
M.OIL(C24-C40)		8426268 ✓	16689.57450 ✓	504.882 ✓
M.OIL(C28-C35)		3201901 ✓	6979.25356 ✓	458.774 ✓
Totals:		45632478		2476.488



*Act*  
 05/25/13

Software Version: Version 3.3.1

**Figure 2C: TYPICAL n-ALKANE CHROMATOGRAM**

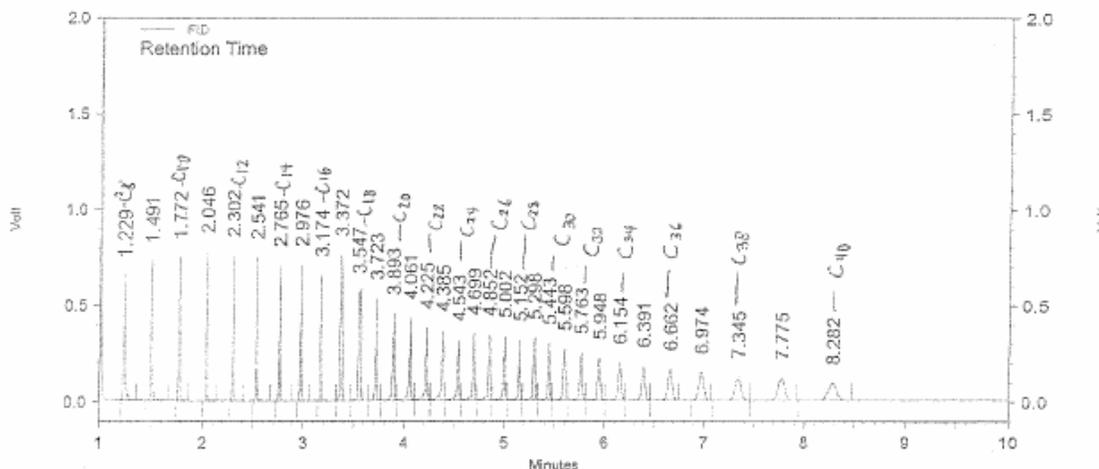
METHOD 8015 by GC/FID  
 EMAX Laboratories, Inc.

Inst. Name: : GCT-105 (Offline)  
 File : D:\Projects\EZC331\Data\LE24\LE24019.dat  
 Method : D:\Projects\EZC331\Method\DSD5E24.met  
 Sequence: : D:\Projects\EZC331\Sequence\LE25.seq  
 Sample ID : DRO(C8-C40 + C9-C39)  
 Acquired : 05/24/13 16:49:57  
 Printed : 05/24/13 17:24:54  
 User : CHERRY

**FID Results**

Name	Retention Time	Area	Average RF	ESTD conc. [ ppm ]
BROMOBENZENE				0.000 BDL
HEXACOSANE	4.852	453502	24896.70051	18.215
DIESEL(TOTAL)		17466067	31462.71743	555.135
DIESEL(C10-C24)		8963628	30401.78600	294.839
DIESEL(C10-C28)		10713865	30544.07919	350.767
DIESEL(C10-C25)		9387897	30519.36600	307.605
DIESEL(C9-C24)		8963628	30968.89186	289.440
DIESEL(C9-C25)		9387897	31086.47186	301.993
DIESEL(C10-C36)		13717177	30544.88929	449.083
DIESEL(C10-C40)		16176133	30544.88929	529.586

Totals		95229794		3096.662
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Software Version: Version 3.3.1

*As*  
 05/28/13

**Figure 3A: TYPICAL DIESEL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD M8015

Lab Name : EMAX Inc  
 Instrument ID : D5  
 GC Column : HP5  
 Column size ID : 30MX0.32MM 0.25UM  
 LFID & Datetime: LE24003A 05/24/13 11:45  
 LFID & Datetime: LE24004A 05/24/13 12:02  
 LFID & Datetime: LE24005A 05/24/13 12:19  
 LFID & Datetime: LE24006A 05/24/13 12:36  
 LFID & Datetime: LE24007A 05/24/13 12:53  
 LFID & Datetime: LE24008A 05/24/13 13:10  
 LFID & Datetime: LE24009A 05/24/13 13:26  
 CONC UNIT: ppm

COMPOUND	CONC X	CALIBRATION FACTORS						(AREA)/UNIT		MEAN	%RSD
		1.00X	2.00X	10.00X	20.00X	100.00X	300.00X	600.00X			
DIESEL(TOTAL)	5.00	38471	26569	31612	31841	32719	30432	28596	31462.7	11.9	
DIESEL(C10-C24)	5.00	37734	25739	30612	30775	31457	29131	27365	30401.8	12.6	
DIESEL(C10-C28)	5.00	37974	25911	30708	30870	31604	29288	27455	30544.1	12.6	
DIESEL(C10-C25)	5.00	37974	25911	30691	30849	31556	29235	27419	30519.4	12.6	
DIESEL(C9-C24)	5.00	37734	26114	31302	31461	32182	29882	28106	30968.9	11.8	
DIESEL(C9-C25)	5.00	37974	26285	31382	31536	32281	29987	28160	31086.5	11.9	
DIESEL(C10-C36)	5.00	37974	25911	30708	30870	31604	29290	27458	30544.9	12.6	
DIESEL(C10-C40)	5.00	37974	25911	30708	30870	31604	29290	27458	30544.9	12.6	
SURROGATE	X	0.00X	1.00X	2.00X	3.00X	4.00X	5.00X	11.00X	MEAN	%RSD	
BROMOBENZENE	20.00	0	17693	18001	17396	19578	20967	20590	19037.3	8.1	
HEXACOSANE	5.00	0	25121	24531	24139	25211	25650	24728	24896.7	2.2	

DSD5E24.MET

**Figure 3B: TYPICAL JP5 & MOTOR OIL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD M8015

Lab Name : EMAX Inc  
 Instrument ID : 05  
 GC Column : HP5  
 Column size ID : 30MX0.32MM-0.25UM  
 LFID & Datetime: LE24011A 05/24/13 14:00  
 LFID & Datetime: LE24012A 05/24/13 14:17  
 LFID & Datetime: LE24013A 05/24/13 14:34  
 LFID & Datetime: LE24014A 05/24/13 14:51  
 LFID & Datetime: LE24015A 05/24/13 15:08  
 LFID & Datetime: LE24016A 05/24/13 15:25  
 CONC UNIT: ppm

COMPOUND	CONC X	CALIBRATION FACTORS						MEAN	%RSD
		2.00X	10.00X	20.00X	100.00X	300.00X	600.00X		
JP5(C8-C18)	5.00	30671	32520	33471	32088	30498	30405	31608.8	4.0
M.OIL(C18-C36)	5.00	15584	20219	21712	20462	19448	19955	19563.2	10.7
M.OIL(C24-C36)	5.00	13829	17322	18567	17099	16152	16669	16606.4	9.5
M.OIL(C24-C40)	5.00	13829	17322	18567	17099	16375	16945	16689.6	9.5
M.OIL(C28-C35)	5.00	6354	7008	7632	6929	6639	7313	6979.3	6.6

DSD5E24.MET

**Figure 4A: TYPICAL DIESEL CONTINUING CALIBRATION SUMMARY**

CONTINUE CALIBRATION  
 METHOD M8015

Lab Name : EMAX Inc  
 Instrument ID : D5  
 GC Column : HP5  
 Column size ID : 30MX0.32MM 0.25UM  
 Mid Conc Init LFID & Datetime: LE24007A 05/24/2013 12:53  
 Conc Cont LFID & Datetime: LH23004A 08/23/2013 10:04  
 CONC UNIT : ppm

COMPOUND	RT MINUTES	RT WINDOW		TRUE CONC	AVERAGE CF	RESULT			QL	%D LIMITS
		FROM	TO			AREA	CONC	%D		
DIESEL(TOTAL)	NA	NA	NA	500.0	31462.7	18083552	574.76	15		20
DIESEL(C10-C24)	NA	NA	NA	500.0	30401.8	17063556	561.27	12		20
DIESEL(C10-C28)	NA	NA	NA	500.0	30544.1	17138532	561.11	12		20
DIESEL(C10-C25)	NA	NA	NA	500.0	30519.4	17118376	560.90	12		20
DIESEL(C9-C24)	NA	NA	NA	500.0	30968.9	17662380	570.33	14		20
DIESEL(C9-C25)	NA	NA	NA	500.0	31086.5	17717200	569.93	14		20
DIESEL(C10-C36)	NA	NA	NA	500.0	30544.9	17138532	561.09	12		20
DIESEL(C10-C40)	NA	NA	NA	500.0	30544.9	17138532	561.09	12		20
SURROGATE	MINUTES	FROM	TO	TRUECON	CF	AREA	CONC	%D	QL	LIMITS
BROMOBENZENE	1.584	1.581	1.587	80.0	19037.3	1605353	84.33	5		20
HEXACOSANE	4.893	4.845	4.941	20.0	24896.7	583311	23.43	17		20

**Figure 4B: TYPICAL JP5 & MOTOR OIL CONTINUING CALIBRATION SUMMARY**

CONTINUE CALIBRATION  
 METHOD M8015

Lab Name : EMAX Inc  
 Instrument ID : D5  
 GC Column : HP5  
 Column size ID : 30MX0.32MM 0.25UM  
 Mid Conc Init LFID & Datetime: LE24014A 05/24/2013 14:51  
 Conc Cont LFID & Datetime: LH23005A 08/23/2013 10:21  
 CONC UNIT : ppm

COMPOUND	RT MINUTES	RT WINDOW		TRUE CONC	AVERAGE CF	RESULT			QL	%D LIMITS
		FROM	TO			AREA	CONC	%D		
JP5(C8-C18)	NA	NA	NA	500.0	31608.8	16151035	510.97	2		20
M.OIL(C18-C36)	NA	NA	NA	500.0	19563.2	10229411	522.89	5		20
M.OIL(C24-C36)	NA	NA	NA	500.0	16606.4	8612622	518.63	4		20
M.OIL(C24-C40)	NA	NA	NA	500.0	16689.6	8623910	516.72	3		20

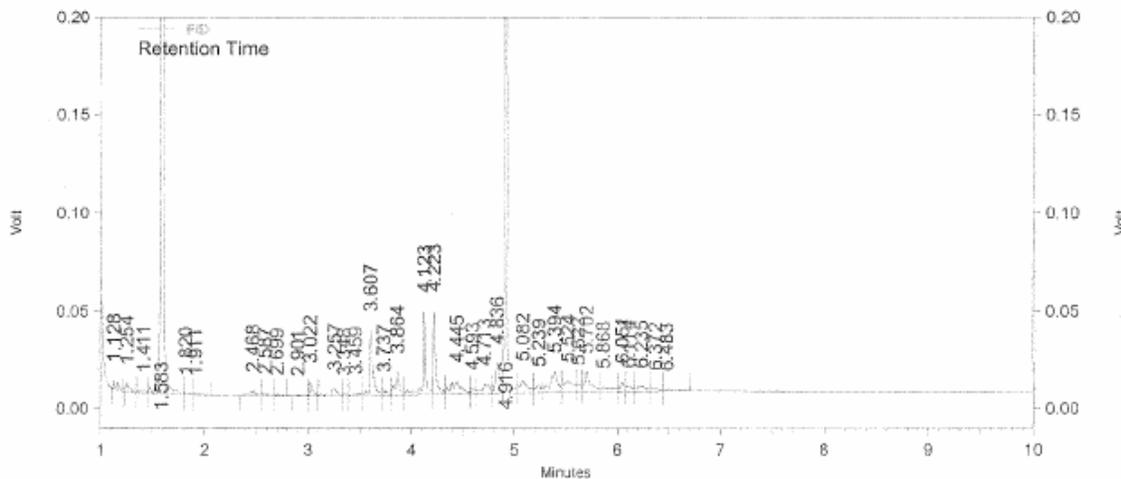
**Figure 5: TYPICAL RAW DATA**

METHOD 8015 by GC/FID  
 EMAX Laboratories, Inc.

Inst. Name: : GCT-105 (Offline)  
 File : D:\Projects\EZC331\Data\LH23\LH23009.dat  
 Method : D:\Projects\EZC331\Method\DSD5E24M.met  
 Sequence: : D:\Projects\EZC331\Sequence\LH23.seq  
 Sample ID : 13H147-01  
 Acquired : 08/23/13 11:54:27  
 Printed : 08/29/13 16:57:40  
 User : KYAW

**FID Results**

Name	Retention Time	Area	Average RF	ESTD conc. [ ppm ]
BROMOBENZENE	1.583	1480743	19037.34242	77.781
HEXACOSANE	4.916	667467	24896.70051	26.809
DIESEL(TOTAL)		721635	31462.71743	22.936
DIESEL(C10-C24)		351944	30401.78600	11.576
M.OIL(C24-C36)		341625	16606.39789	20.572
<b>Totals</b>		<b>3563414</b>		<b>159.675</b>



Software Version: Version 3.3.1

**Figure 6: TYPICAL SAMPLE RESULT SUMMARY**

METHOD SW3550B/SW8015C  
 PETROLEUM HYDROCARBONS BY EXTRACTION

```

=====
Client       : XYZ, INC.                Date Collected: 08/15/13
Project      : CLEAN LAND              Date Received: 08/16/13
Batch No.    : 13H147                  Date Extracted: 08/22/13 16:51
Sample ID    : IDW-235                 Date Analyzed: 08/23/13 11:54
Lab Samp ID  : H147-01                 Dilution Factor: 1
Lab File ID  : LH23009A                Matrix          : SOIL
Ext Btch ID  : DSH040S                 % Moisture      : 2.5
Calib. Ref.  : LH23004A                Instrument ID   : GCT105
=====
  
```

PARAMETERS	RESULTS (mg/kg)	LOQ (mg/kg)	LOD (mg/kg)
DIESEL	12	10	5.1
MOTOR OIL	21	10	5.1

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
BROMOBENZENE	79.8	102.6	77.8	50-130
HEXACOSANE	27.5	25.64	107	60-140

RL : Reporting Limit  
 Parameter H-C Range  
 Diesel C10-C24  
 Motor oil C24-C36

Figure 7:

TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND  
 BATCH NO.: 13H147  
 METHOD: METHOD SW3550B/SW8015C

MATRIX: SOIL % MOISTURE: NA  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: MBLK1S  
 LAB SAMP ID: DSH0405B DSH0405L DSH0405C  
 LAB FILE ID: LH23006A LH23007A LH23008A  
 DATE EXTRACTED: 08/22/1316:51 08/22/1316:51 08/22/1316:51 DATE COLLECTED: NA  
 DATE ANALYZED: 08/23/1311:03 08/23/1311:20 08/23/1311:37 DATE RECEIVED: 08/22/13  
 PREP. BATCH: DSH0405 DSH0405 DSH0405  
 CALIB. REF: LH23004A LH23004A LH23004A

ACCESSION:

PARAMETER	BLNK RSLT (mg/kg)	SPIKE AMT (mg/kg)	BS RSLT (mg/kg)	BS % REC	SPIKE AMT (mg/kg)	BSD RSLT (mg/kg)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Diesel	ND	500	409	82	500	417	83	2	60-150	30

SURROGATE PARAMETER	SPIKE AMT (mg/kg)	BS RSLT (mg/kg)	BS % REC	SPIKE AMT (mg/kg)	BSD RSLT (mg/kg)	BSD % REC	QC LIMIT ( % )
Bromobenzene	100	88.1	88	100	77.4	77	50-130
Hexacosane	25.0	25.4	102	25.0	21.7	87	60-140

**Figure 8: TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND  
 BATCH NO.: 13H147  
 METHOD: METHOD SW3550B/SW8015C

=====

MATRIX:	SOIL			% MOISTURE:	2.5
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	IDW-235				
LAB SAMP ID:	H147-01R	H147-01M	H147-01S		
LAB FILE ID:	LH23009A	LH23010A	LH23011A		
DATE EXTRACTED:	08/22/1316:51	08/22/1316:51	08/22/1316:51	DATE COLLECTED:	08/15/13
DATE ANALYZED:	08/23/1311:54	08/23/1312:20	08/23/1312:35	DATE RECEIVED:	08/16/13
PREP. BATCH:	DSH040S	DSH040S	DSH040S		
CALIB. REF:	LH23004A	LH23004A	LH23004A		

ACCESSION:

PARAMETER	SMPL RSLT (mg/kg)	SPIKE AMT (mg/kg)	MS RSLT (mg/kg)	MS % REC	SPIKE AMT (mg/kg)	MSD RSLT (mg/kg)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Diesel	21	546	525	92	546	544	96	4	50-140	30

SURROGATE PARAMETER	SPIKE AMT (mg/kg)	MS RSLT (mg/kg)	MS % REC	SPIKE AMT (mg/kg)	MSD RSLT (mg/kg)	MSD % REC	QC LIMIT (%)
Bromobenzene	109	100	92	109	97.9	90	50-150
Hexacosane	27.3	23.9	88	27.3	22.9	84	50-150

Figure 9:

TYPICAL CASE NARRATIVE

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN LAND  
SDG : 13H147

METHOD SW3550B/SW8015C  
PETROLEUM HYDROCARBONS BY EXTRACTION

One (1) soil sample was received on 08/16/13 for TPH analysis, Method SW3550B/SW8015C in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

The sample was analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for DCH040SL/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the sample in this SDG. Percent recoveries for H147-01M/S were within project QC limits.

Surrogate

Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

The sample was analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURES**

QC Procedure	Frequency	Acceptance Criteria	Corrective Action	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Minimum five-point initial calibration	Initially; as needed	Linear - mean RSD ≤ 20%	Correct the problem then repeat initial calibration		
Second-source calibration verification	After initial calibration	Within ± 25% of expected value	Correct the problem then repeat initial calibration		
Initial calibration verification	Daily, before sample analysis	Within ± 20% of expected value	Correct the problem then repeat initial calibration		
Calibration verification	Every 12 hours of analysis time and at the end of analysis sequence	Within ± 20% of expected value	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification		
Method Blank (MB)	One MB per preparation batch	No analyte detected > ½ LOQ	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
Lab Control Sample (LCS)	One LCS per preparation batch	Within EMAX QC Limits	Re-prep and re-analyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard, and method blank	Within EMAX QC Limits	Correct the problem then re-extract and re-analyze sample		
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 project samples per matrix	Refer to EMAX QC Limits	None		
Chromatogram	All sample results	Within calibration range  NO SATURATED PEAK(s)	Dilute and re-analyze all samples over the calibration range  Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range.		
<b>Comments:</b> Refer to PSR for flagging criteria.			Reviewed By:		
			Date:		

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
 DIESEL  
 METHOD: SW 8015C**

Conc Unit mg/Kg  
 Sample Amount(g): 10  
 Volume Extracted(ml): 10

Date Extracted: 11/5 & 11/12/12  
 Extracted by: C. Siu  
 Date Analyzed: 11/13 & 11/5/12  
 Analyzed by: K. Linn

PARAMETER	LK05034A	LK05035A	LK13006A	LK13007A	TV	Ave. Conc., mg/L	Ave. %Rec	SD	RSD	QC Criteria	Comments
	DSK009SL	DSK009SC	DSK021SL	DSK021SC							
Total Diesel	446	397	501	475	500	455	91	44.53	9.80	70 - 130	Passed
Diesel(C <sub>10</sub> -C <sub>24</sub> )	451	403	494	470	500	455	91	38.71	8.52	70 - 130	Passed
Bromobenzene	86.7	82.4	92.0	88.6	100	87	87	3.99	4.56	60 - 130	Passed
Hexacosane	19.3	18.6	21.0	20.5	25	20	79	1.09	5.48	60 - 130	Passed









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SOP REVIEW FORM

EMAX-8015G  
SOP No.

Rev. 5  
Revision Number

GASOLINE RANGE ORGANICS  
Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

No update / revision required

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

*Fatima Madamba*  
FATIMA MADAMBA

Date:

7/11/15

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**

SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Prepared By: Lucita Arzadon *L.A. Arzadon* Date: 01-24-14

Approved By: Kenette Pimentel *K. Pimentel* Date: 01-24-14  
QA Manager

Approved By: Caspar Pang *C. Pang* Date: 01-24-14  
Laboratory Director

Control Number: **8015G-05-****1.0 SCOPE AND APPLICATION**

- 1.1. This method is applicable for analyzing purgeable petroleum hydrocarbons bracketed by the range of alkanes from C<sub>6</sub> to C<sub>10</sub> as gasoline range organics (GRO) in samples of various matrices (i.e. soils, water, sludge). Other range of alkanes may be applied (e.g., C<sub>5</sub> to C<sub>10</sub>, C<sub>6</sub> to C<sub>12</sub>, etc.) or other purgeable fuel patterns such as JP4 and Stoddard may be analyzed provided that qualitative identification and quantitative determination is properly established.
- 1.2. This SOP is an adaptation of SW846 Method 8015C. This SOP is also applicable to SW846 Method 8015B and Method 8015D.

**2.0 SUMMARY OF METHOD**

- 2.1. A known amount of sample is purged by inert gas into a trap and retains the purgeable organic compounds. The trap is back flushed into the GC system equipped with flame ionization detector (FID). The instrument is calibrated with a gasoline standard. Hydrocarbon markers (C<sub>5</sub> to C<sub>12</sub>) are analyzed to determine elution time. Quantitation of typical GRO is based on C<sub>6</sub> to C<sub>10</sub> alkane range.
- 2.2. **Interferences**
- 2.2.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to use.
- 2.2.2. Carry-over from a highly concentrated sample can be a potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.

**3.0 DETECTION LIMITS****3.1. Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Established DL, LOD and LOQ are as follows:

Analyte	Water (µg/L)			Soil 1 gm to 5 mL (µg/Kg)			Soil 5 gm to 5 mL MeOH (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ	DL	LOD	LOQ
Gasoline	5	10	20	20	40	100	350	500	1000
JP4	8.4	10	20	-	-	-	250	500	1000

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Analyte	Water ( $\mu\text{g/L}$ )			Soil 1 gm to 5 mL ( $\mu\text{g/Kg}$ )			Soil 5 gm to 5 mLMeOH ( $\mu\text{g/Kg}$ )		
	DL	LOD	LOQ	DL	LOD	LOQ	DL	LOD	LOQ
Stoddard	10	20	50	-	-	-	250	500	1000
Bromofluorobenzene	2.5	5	20	10	20	100	50	100	1000
1,1,1-Trifluorotoluene	2.5	5	20	10	20	100	50	100	1000

**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (See Section 9.3.1). Dilute and re-analyze all samples having results above this range for proper quantitation.
- 4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (See Section 9.3.1). Lower the dilution factor and re-analyze all diluted samples analyzed below this range for proper quantitation.

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1. Holding Time****5.1.1. Aqueous Samples**

- 5.1.1.1. Analyze preserved samples within 14 days from sampling date.
- 5.1.1.2. If pH > 2, analyze samples within 7 days from sampling date.

**5.1.2. Soil Samples**

- 5.1.2.1. Samples received in encore are extracted with methanol within 48 hours from sampling time and analyzed within 14 days from sampling date unless otherwise specified by the project.
- 5.1.2.2. Sample received in jars are extracted with methanol and analyzed within 14 days from sampling date.
- 5.1.2.3. Analyze soil samples (1gram direct purge) within 14 days from sampling date.

**5.2. Preservation**

- 5.2.1. Water samples received are expected to be contained in 40 ml vial with teflon-lined septa preserved at pH < 2 with HCl with zero head space.
- Note: The size of any bubble caused by degassing upon cooling the sample should not exceed 6 mm.<sup>1</sup>
- 5.2.2. Store samples and extracts at  $\leq 6^{\circ}\text{C}$ .

<sup>1</sup> Referenced from SW846 Method 5030C, Section 8.1.

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-5030 Purge and Trap
- 6.2. EMAX-5035 Purge and Trap, Closed System
- 6.3. EMAX-DM01 Data Flow and Review
- 6.4. EMAX-QA04 Detection Limit
- 6.5. EMAX-QA05 Training
- 6.6. EMAX-QA08 Corrective Action
- 6.7. EMAX-QC01 Quality Control of Chemicals
- 6.8. EMAX-QC02 Analytical Standard Preparation
- 6.9. EMAX-QC07 Glassware Cleaning
- 6.10. EMAX-SM03 Waste Disposal
- 6.11. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all SDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS & REAGENTS****8.1. Instruments and Supplies**

Purge and Trap System (Concentrator)	LSC 2000 Tekmar (GC39)/OI4560 or equivalent (GC55)
Autosampler	Archon or equivalent
Gas Chromatography	HP 5890 Series II, GC with FID
Column	DB5 – 30m x .53 mm, 1.5 µm thickness, or equivalent
Gas	ultra-high purity helium, ultra-high purity hydrogen, hydrogen generator unit, compressed air
Syringes	5 ml Luerlok hypodermic gas-tight
Microsyringes	Hamilton or equivalent
Data System	EZ-Chrom

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Purge Trap	Supelco, Trap "G" or equivalent
------------	---------------------------------

**8.2. Chemicals and Reagents**

- 8.2.1. Methanol, purge and trap
- 8.2.2. Organic-free Reagent Water
- 8.2.3. Organic-free Ottawa Sand

**9.0 STANDARDS****9.1. Stock Standard**

9.1.1. Purchase stock standards or equivalent as certified solutions traceable to NIST standards.

Name	Source	Solvent	Conc. (µg/mL)	Intended Use
Unleaded Gas Comp. Standard	Restek	Methanol	2,500	Calibration
Stoddard Solvent Standard	Restek	Methanol	10,000	Calibration
JP4 Standard	Restek	Methanol	50,000	Calibration
Cert. BTEX Unleaded Gas	AccuStandard	Methanol	5,000	ICV/LCS/MS
Stoddard Solvent	AccuStandard	Methanol	20,000	ICV/LCS/MS
JP4 Jet Fuel	AccuStandard	Methanol	20,000	ICV/LCS/MS

**9.2. Intermediate Standards**

- 9.2.1. Intermediate and working standards are prepared according to EMAX-QC02.
- 9.2.2. All standards should be transferred in inert vials labeled with ID from standard preparation logbook, date of expiration, concentration, and stored with minimal headspace at -10°C to -20°C.
- 9.2.3. Intermediate standards are prepared only for Stoddard and JP4 as follows:

Stock Standard			Solvent	Intermediate Standard	
Standard Name	Conc. (µg/ml)	Aliquot (µl)		Final Vol. (ml)	Final Conc. (mg/L)
Stoddard Solvent (1 <sup>st</sup> source)	10,000	500	Methanol	2	2,500
Stoddard Solvent (2 <sup>nd</sup> source)	20,000	250	Methanol	2	2,500
JP4 Standard (1 <sup>st</sup> source)	50,000	100	Methanol	2	2,500
JP4 Standard (2 <sup>nd</sup> source)	20,000	250	Methanol	2	2,500

**9.3. Calibration Standards**

- 9.3.1. Initial Calibration Standards
  - 9.3.1.1. Gasoline

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot ( $\mu$ l)		Final Conc. ( $\mu$ g/L) Gasoline /Surrogate
	Gasoline Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30
4	1	2	500/40
5	2	2.5	1000/50
6	4	3.75	2000/75
7	5 $\mu$ l	5	2500/100

9.3.1.2. Stoddard

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot ( $\mu$ L)		Final Conc. ( $\mu$ g/L) Stoddard / Surrogate
	Stoddard Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30
4	1	2	500/40
5	2	2.5	1000/50
6	3	3	1500/60

9.3.1.3. JP4

Prepare initial calibration standards in 5 ml organic- free water with concentrations as suggested below:

ICAL Standard	Aliquot ( $\mu$ L)		Final Conc. ( $\mu$ g/L) JP4/ Surrogate
	JP4 Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
1	0.04	0.5	20/10
2	0.1	1	50/20
3	0.2	1.5	100/30

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

ICAL Standard	Aliquot (µL)		Final Conc. (µg/L) JP4/ Surrogate
	JP4 Std. (2,500 mg/L)	Surrogate BFB/TFT (100 mg/L)	
4	1	2	500/40
5	2	2.5	1000/50
6	4	3.75	2000/75
7	5	5	2500/100

9.3.2. Initial Calibration Verification Standards

Prepare initial calibration verifications standards (using second-source standards) in 5 ml organic-free water with concentrations as suggested below:

ICV Standard	Intermediate Std. (2 <sup>nd</sup> source)		Surrogate BFB/TFT (100 mg/L) Aliquot in µL	Final Conc. (µg/L) Analyte / Surrogate
	Concentration (mg/L)	Aliquot (µL)		
Gasoline	5000	1	2.5	1000/50
Stoddard	2500	2	2.5	1000/50
JP4	2500	2	2.5	1000/50

9.3.3. Continuing Calibration Standard

Using intermediate standards, prepare daily calibration standards in a syringe with 5 ml of organic-free water as suggested below:

DCC Standard	Intermediate Std. (1 <sup>st</sup> source)		Surrogate BFB/TFT (100 mg/L) Aliquot in µL	Final Conc. (µg/L) Analyte / Surrogate
	Concentration (mg/L)	Aliquot (µL)		
Gasoline	2500	2	2.5	1000/50
Stoddard	2500	2	2.5	1000/50
JP4	2500	2	2.5	1000/50

9.4. **LCS/Matrix Spike Standard**

9.4.1. Use second-source intermediate standards for laboratory control standard (LCS) and matrix spike (MS) standards. Refer to Section 9.2.3 for Stoddard and JP4. Use Gasoline standard from AccuStandard (5,000 mg/L) for gasoline spike.

9.5. **Surrogate Standards**

9.5.1. Purchase stock standards or equivalent as certified solutions traceable to NIST standards.

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Name	Source	Solvent	Conc. (µg/mL)
Bromofluorobenzene / TFT	Ultra Scientific	Methanol	2000

9.5.2. Prepare intermediate surrogate standard as follows:

Stock Standard			Solvent	Intermediate Standard	
Standard Name	Conc. (µg/ml)	Aliquot (µl)		Final Vol. (ml)	Final Conc. (mg/L)
Bromofluorobenzene / TFT	2000	100	Methanol	2	100

9.6. **Retention Time Window Standard**

9.6.1. Purchase the following standards or equivalent.

Name	Source	Conc. (mg/L)
GRO	AccuStandard	2000
2-Methylpentane	Supelco	2000
1,2,4-Trimethylbenzene	Ultra Scientific	5000 *
DRO	AccuStandard	2000

\* Dilute to 2000 mg/L before use for retention time analysis.

9.7. Other concentration levels may be used as appropriate.

**10.0 PROCEDURES****10.1. Sample Preparation**

- 10.1.1. Prepare aqueous samples in accordance with EMAX-5030. Add surrogates to all samples to yield 40µg/L prior to purging unless otherwise specified by the project.
- 10.1.2. Prepare soil samples in accordance with EMAX-5035. Add surrogates to all samples to yield 40µg/Kg prior to purging unless otherwise specified by the project.
- 10.1.3. Prepare LCS/LCD by spiking a reagent blank to yield 500 µg/L spike standard unless otherwise specified by the project. Add surrogate at the same level as the samples.
- 10.1.4. Prepare MS/MSD sample using the assigned matrix QC sample similarly as the LCS sample.

**10.2. Instrument Parameters**

10.2.1. Initially, set the instrument parameters as suggested in the table below.

10.2.2. Gas Chromatographic Condition

Carrier gas flow (column) helium	9-10 ml/min
Make up gas (He)	20-21 ml/min

## STANDARD OPERATING PROCEDURES

**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14

Air	60 psi
Helium	80 psi
Hydrogen	50 psi

<b>Temperature Settings:</b>					
<b>Inst. GC 39</b>			<b>Inst. GC 55</b>		
Injector Temperature:		OFF	Injector Temperature:		150 °C
Detector Temperature:		235 °C	Detector Temperature:		235 °C
Temp °C	Hold Time,min	Rate, °C/min	Temp °C	Hold Time,min	Rate, °C/min
35	6	0	35	6	0
70	0	8	70	0	8
120	3	5	120	3	5
245	4	30	245	3	30

10.2.3. Purge and Trap Condition

	<b>Inst. GC 39</b>	<b>Inst. GC 55</b>
Purge	10 mins at 40 °C	15 mins at 30 °C
Dry Purge / Desorb Preheat	3 mins at 170 °C	1 min at 180 °C
Desorb	2 mins at 180 °C	2 mins at 180 °C
Bake	14 mins at 185 °C	10 mins at 220 °C

10.2.4. Optimize the instrument for its intended use.

10.2.5. Record the instrument operating condition on the instrument maintenance log and post the latest instrument parameter setup in front of the instrument for ease when performing the instrument routine check.

10.2.6. When instrument parameter setup requires change due to instrument optimization document the change as described in Section 10.2.5.

10.3. **Calibration**10.3.1. Initial Calibration (ICAL)

10.3.1.1. Analyze initial calibration standards (Refer to Section 9.3.1 for standard preparation) as described in Section 10.4.

10.3.1.2. Refer to Section 10.5 for calculation.

10.3.1.3. Acceptance criteria are specified in Appendix 1.

10.3.2. Initial Calibration Verification (ICV)

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10.3.2.1. After establishing ICAL, analyze the ICV standard (Refer to Section 9.3.2 for standard preparation) to verify the validity of the ICAL. Refer Appendix 1 for acceptance criteria. If non-compliant refer to Section 12 for corrective action.

10.3.3. Retention Time Window Check (RTW)

10.3.3.1. Spike 5 ml water reagent with 0.5 µL of 2000 mg/L RTW standard.

10.3.3.2. Analyze the RTW standard after every ICAL to set carbon cut-off ranges (e.g, C<sub>6</sub> - C<sub>10</sub>, C<sub>5</sub> - C<sub>12</sub>). Refer to Section 10.4.3.

10.3.4. Continuing Calibration (DCC)

10.3.4.1. Analyze the DCC standard (Refer to Section 9.3.3 for standard preparation) to verify the validity of the ICAL. Refer to Appendix 1 for acceptance criteria. If non-compliant refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Analyze instrument blank to ensure that the instrument is free from contamination.

10.4.1.2. Analyze DCC to check ICAL validity.

10.4.1.3. Analyze Method Blank to check for preparation batch contamination.

10.4.1.4. Analyze Lab Control Sample to check accuracy.

10.4.1.5. Analyze Lab Control Sample Duplicate (if required by the project).

10.4.1.6. Analyze samples to a maximum number of 12-hour runs or as specified by the project.

10.4.1.7. Analyze matrix spikes (MS/MSD) per project requirement.

10.4.1.8. Record the analytical sequence in the Analytical Run Log.

10.4.1.9. Print instrument sequence before and after the analysis run and attach to the analytical run. Document any changes that occurred during the process.

10.4.2. Identification and Quantitation

10.4.2.1. Identification is based on pattern recognition. Hence, compare sample chromatograms to reference hydrocarbons standard chromatograms for their response hydrocarbon range and peak distribution to determine the most probable petroleum product.

10.4.2.2. All peaks eluting within the established RT window identifies the GRO, JP4 and Stoddard.

10.4.2.3. When the elution profile of a sample does not match that of gasoline standard, JP4 or Stoddard, but falls within the retention time window, quantitate results as gasoline range organics (GRO) and denote the observed deviation in case narrative.

10.4.2.4. Quantitation is achieved by the summation of all peaks in the chromatogram minus the solvent peak, and the sample result is calculated using Equation 10.5.3.

10.4.2.5. Integrate the total peak area response and quantitate the total area by using the ACF of Gasoline, JP4 or Stoddard (see section 10.5.1).

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10.4.2.6. When manual integration is necessary follow the procedures described in EMAX-DM01 Section 4.4.3.

**10.4.3 Retention Time Windows (RTW)****10.4.3.1 Establishing RTW**

10.4.3.1.1 Run RTW standard over a period of 72 hours.

10.4.3.1.2 Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte (Use Equation 10.5.1.2).

10.4.3.1.3 The width of RTW is defined by  $\pm 3XSD$  obtained from Section 10.4.3.1.2.

**10.4.3.2 Evaluating RTW**

10.4.3.2.1 If the SD is equal to 0.00, default to the previous study until historical data is obtained to define the RTW for the current instrument.

10.4.3.2.2 For new instruments, use the established retention time from another instrument having the same instrument parameters (e.g. detector, temperature program and column.) If there are no instruments with the same instrument parameter, use 0.03 minutes as the default RTW until historical data is obtained to define the RTW for the current instrument parameters condition.

**10.4.3.3 Application of RTW**

10.4.3.3.1 Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.3.3.2 Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

**10.4.3.4 Updating RTW**

10.4.3.4.1 Re-establish the RTW as described in Section 10.4.3.1 when any of the following conditions occur.

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of the RTW)
- Major instrument maintenance (e.g. replacements of detector or column, temperature program change, etc.)

10.4.3.4.2 If the calculated new RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

**10.4.4 Sample Result Evaluation**

10.4.4.1 Check surrogate recoveries against project specific requirement (PSR). In the absence of PSR, default to in-house QC procedures described in Appendix 1.

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- 10.4.4.2 Dilute and re-analyze samples having concentrations greater than the highest calibration range.
- 10.4.4.3 Dilute and re-analyze samples having saturated peak(s) within C<sub>6</sub> – C<sub>10</sub>. See Figure 1 for typical saturated peak.
- 10.4.4.4 Re-analyze samples suspected of carry-over from a proceeding sample that has high concentration.
- 10.4.4.5 Report discrete peak(s) observed as required by the project. Column bleed subtraction is not generally required in GRO analysis.

**10.5 Calculations****10.5.1 Initial Calibration****10.5.1.1 Calculate the Calibration Factor (CF)**

$$CF = \frac{R_a}{C_k} \quad \text{Eq.-10.5.1.1}$$

where:

*CF* - is the calibration factor

*R<sub>a</sub>* - is the analyte response measured in peak area

*C<sub>k</sub>* - is the known concentration of the analyte in µg/L (water); µg/Kg (soil)

**10.5.1.1 Calculate the Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}} \quad \text{Eq.-10.5.1.2}$$

where:

*SD* - is the standard deviation

*x<sub>i</sub>* - is the result at the *i*<sup>th</sup> measurement

$\bar{x}$  - is the mean

*N* - is the number of measurements

**10.5.1.1 Calculate the Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \left[ \frac{SD}{ACF} \right] 100 \quad \text{Eq.-10.5.1.3}$$

where:

*%RSD* - is the percent relative standard deviation

*SD* - is the standard deviation

*ACF* - is the average calibration factor

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**GASOLINE RANGE ORGANICS**SOP No.: EMAX-8015G Revision No. 5 Effective Date: 24-Jan-14**10.5.1.1 Calculate the Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF}{N} \quad \text{Eq.-10.5.1.4}$$

*where:**ACF - is the average calibration factor* *$\sum CF$  - is the summation of the calibration factors**N - is the number of calibration points***10.5.2 Calculate the Percent Difference for DCC from ACF**

$$\%D = \frac{|C_k - C_f|}{C_k} * 100 \quad \text{Eq.-10.5.2}$$

*where:**%D - is the percent difference DCC from the ACF**C<sub>k</sub> - is the known concentration of analyte, in µg/L**C<sub>f</sub> - is the concentration found, in µg/L***10.5.3 Calculate Sample Results****10.5.3.1 Water Samples**

$$C = \left[ \frac{R_a}{ACF} \right] \left[ \frac{V_e}{S_a} \right] DF \quad \text{Eq.-10.5.3.1}$$

**10.5.3.1 Soil Samples**

$$C = \left[ \frac{R_a}{ACF} \right] \left[ \frac{V_e}{(S_a)(\%S)} \right] DF \quad \text{Eq.-10.5.3.2}$$

*where:**C - is the concentration of analyte in µg/L (water), µg/Kg (soil)**R<sub>a</sub> - is the analyte response measured in peak area**ACF - is the average calibration factor from initial standard calibration**V<sub>e</sub> - is the purgeable volume in ml**S<sub>a</sub> - is the sample amount in ml (water); g (soil)**DF - is the dilution factor**%S - is the percent solid of the sample***10.5.4 Accuracy and Precision****10.5.4.1 Percent Recovery**

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$$\% R = \left[ \frac{(C_f - C_s)}{C_o} \right] 100 \quad \text{Eq.-10.5.4.1}$$

where:

% - is the percent recovery

$C_f$  - is the concentration found, in  $\mu\text{g/L}$

$C_s$  - is the concentration of the sample in  $\mu\text{g/L}$  (water); in  $\mu\text{g/Kg}$  (soil). For LCS,  $C_s=0$

$C_o$  - is the known concentration of spiked solution

#### 10.5.4.1 Relative Percent Difference

$$\% RPD = \frac{|C_1 - C_2|}{\left( \frac{C_1 + C_2}{2} \right)} \times 100 \quad \text{Eq.-10.5.4.2}$$

where:

RPD - is the relative percent difference

$C_1$  - is the measured concentration of the first sample aliquot

$C_2$  - is the measured concentration of the second sample aliquot

### 10.6 Data Reduction

- 10.6.1 Make a copy of the analytical run log and sample preparation log.
- 10.6.2 Print a copy of the raw data and the QC report.
- 10.6.3 Highlight the data to be reported.
- 10.6.4 Collate the reportable raw data separating the QC results from the sample results.
- 10.6.5 Keep all other data generated with the analytical folder marked with "For record only".

### 10.7 Report Generation

- 10.7.1 Generate the method.txt file using WDBX<sup>2</sup>.exe.
- 10.7.2 Generate the sample results using F1NVX<sup>2</sup>.exe
- 10.7.3 Generate the QC summary using QCVX<sup>2</sup>.exe
- 10.7.4 Generate Lab Chronicle using LABCHRNX<sup>2</sup>.exe
- 10.7.5 Generate the Case Narrative using CN1.exe

### 10.8 Data Review

<sup>2</sup> X – version number of the program.

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- 10.8.1 Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
- Case Narrative
  - Lab Chronicle
  - Sample Results
  - LCS/LCSD Summary
  - MS/MSD Summary
  - ICAL Summary
  - ICV Summary
  - DCC Summary
  - Analytical Log
  - Sample Preparation Log
  - Non-Conformance Report (if any)
- 10.8.2 Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- ✓ Check that qualitative identification is done properly.
  - ✓ Check that surrogate recoveries against Project Specific Requirement (PSR). In the absence of PSR, default to in-house QC limits.
  - ✓ Check that sample results are integrated properly and results over calibration range are diluted and re-analyzed within the calibration range.
  - ✓ Where manual integration was performed, check that it was done properly and documentation was retained in accordance to EMAX-DM01 Section 4.4.3.
  - ✓ Check that presence of saturated peak(s) are diluted, and quantitated properly.
  - ✓ Check that suspected carry-overs are re-analyzed and results are reported accordingly.
  - ✓ Check that discrete peaks [other than column bleeds] are reported according to project requirement.
  - ✓ If any of the above checkpoints indicate a problem, re-analysis is required.
- 10.8.3 Review the attached logs that they are properly filled.
- 10.8.4 Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- 10.8.5 Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.
- 10.8.6 Submit the analytical folder for secondary review.

**10.9 Preventive Maintenance**

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- 10.9.1 On a daily basis, perform the checks listed in the instrument maintenance log prior to sample analysis. Refer to Form 8015GFM – Instrument Maintenance Log.
- 10.9.2 Conduct routine preventive instrument maintenance and document the activity in the instrument-specific maintenance log. Routine maintenance ensures that all equipment are operating under optimum conditions, thus reducing the possibility of instrument malfunction, and consequently affecting sample results. Routine maintenance activities are suggested below:

Maintenance Activity	Description	Frequency
Autosampler	Inspect autosampler needle and check response.	Daily prior to analysis.
Verification	Check gas pressure Check instrument parameters to ensure normal operating conditions. Check instrument performance (e.g., Daily calibration check, instrument blank)	Daily prior to analysis.
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
System Cleaning	Remove dust from fans and vent covers. Lubricate mechanical parts.	Every 6 months or as necessary
Check Flow Path Components	Change the carrier gas trap(s) and purifier	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting. Replace column if necessary. Replace worn out parts.	Once a year

- 10.9.3 Maintain an inventory of instrument parts and supplies for routine maintenance.

**11.0 QUALITY CONTROL****11.1. Sample Preparation**

- 11.1.1. A preparation batch shall consist of a MB, LCS, MS/MSD and ≤ 20 field samples.
- 11.1.2. Decontaminate volumetric flasks used for standard preparation with methanol.
- 11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use in accordance to EMAX-QC01.

**11.2. Analytical Batch**

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- 11.2.1. Initial Calibration must be established and verified by daily continuing calibration as described in Appendix 1.
  - 11.2.2. Analytical batch shall consist of a valid ICAL, QC samples and field samples bracketed with DCC every 12-hour analytical sequence, unless other frequency is prescribed by the project.
  - 11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing a solvent blank and identifying its result as instrument blank.
  - 11.2.4. Organic-free water shall be used for method blank and LCS for water matrix.
  - 11.2.5. Organic-free sand shall be used for method blank and LCS for soil matrix.

**11.3. Method QC**

- 11.3.1. LOD and LOQ must be established before the analytical procedure can be used and verified according to EMAX-QA04.
- 11.3.2. Retention Time Window must be established and updated as prescribed.
- 11.3.3. Demonstration of capability must be established before the analytical procedure can be used.
- 11.3.4. All analysts conducting this analysis must have established demonstration of capability.

**12.0 CORRECTIVE ACTION**

12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures (Refer to Appendix 1).

**12.2. Calibration**

- 12.2.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.1.1. If RSD > 20%, check each calibration point. If an outlier exists, re-analyze that calibration point.
  - 12.2.1.2. If ICV not within the expected recovery range, review the chromatogram.
    - Bias low results are indicative of poor purging or standard degradation.
    - Bias high is indicative of inaccurate standard injection of instrument or contamination.
    - Consider preparing a fresh ICV standard and re-analyze the ICV.
  - 12.2.1.3. If problem persists, inform the Supervisor prior to re-calibration
- 12.2.2. If the continuing calibration is non-compliant, consider the suggestions described in correcting ICV.
- 12.2.3. If instrument blank/reagent blank is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.3.1. Check the reagent water source, e.g. check if same source was used by a similar analysis on a different instrument to rule out reagent contamination.
  - 12.2.3.2. Bake the sample concentrator and/or GC column for at least 15 min.

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12.2.3.3. Re-calculate the data and/or re-analyze the extract, if any of the above checks reveal a problem.

12.2.3.4. If problem persists, inform the Supervisor prior to re-analysis.

**12.3. Surrogates**

12.3.1. If surrogates are non-compliant, but is not due to matrix effects, consider the following suggestions to correct the problem:

12.3.1.1. Check that the surrogate peak is properly integrated.

12.3.1.2. Check for calculation errors and that the concentrations of the surrogate solutions are correct.

- High recoveries may be due to co-eluting matrix interference, examine the sample chromatogram.
- Low recoveries may be due to a poor purge, check the purge tube with a blank before re-analyzing the sample.

12.3.1.3. Check instrument performance to determine if it is within acceptable guidelines.

**12.4. Sample Preparation QCs**

12.4.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detect, you may report the result upon concurring with the PM.
- If sample results are not reportable, determine the source of contamination and correct the problem. Reanalyze method blank and all samples processed with the contaminated blank.

12.4.2. If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for standard degradation. Prepare a new LCS standard.
- Check instrument performance to determine if it is within acceptable guidelines.
- Reanalyze the extract if any of the above checks reveal a problem.
- Otherwise, re-extract all samples associated with the non-compliant LCS with a new set of QC samples.

12.5. A Non-Conformance Report (NCR) is required when any of the following circumstances occur:

- Anomalies, other than specified in Appendix 1, are observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08 for NCR details.

**13.0 POLLUTION PREVENTION**

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

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**14.0 WASTE MANAGEMENT**

- 14.1. No samples may be dumped on the laboratory sink.
- 14.2. Separate and properly identify all unused expired analytical standards for proper disposal.
- 14.3. Place all wastes generated during analytical process in properly labeled satellite waste containers for proper collection.
- 14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

- 15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Duplicate Sample – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.4. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.6. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.7. Matrix – is a component or form of a sample.
- 15.1.8. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.9. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.10. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

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15.1.11. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

15.1.12. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

**15.2. Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing gasoline range organics unless otherwise other directive is specified by the project requirements.

**15.3. Department of Defense (DoD) and Department of Energy (DOE) Projects**

15.3.1. Samples from DoD and DOE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied

**16.0 REFERENCES**

- 16.1. US EPA SW846 Method 8015C, Revision 3, February 2007.
- 16.2. US EPA SW846 Method 8015D, Revision 4, June 2003.
- 16.3. US EPA SW846 Method 8015B, Revision 2, December 1996.
- 16.4. US EPA SW846 Method 8000B, Revision 2, December 1996.
- 16.5. EMAX Quality Systems Manual, as updated.

**17.0 APPENDICES****17.1. Figures**

- |          |           |  |
|----------|-----------|--|
| 17.1.1.  | Figure 1  | Typical Peak Evaluation                  |
| 17.1.2.  | Figure 2  | Typical GRO Chromatogram                 |
| 17.1.3.  | Figure 3  | Typical Hydrocarbon Marker Chromatograms |
| 17.1.4.  | Figure 4  | Typical ICAL Summary                     |
| 17.1.5.  | Figure 5  | Typical Continuing Calibration Summary   |
| 17.1.6.  | Figure 6  | Typical Raw Data                         |
| 17.1.7.  | Figure 7  | Typical Sample Result Summary            |
| 17.1.8.  | Figure 8  | Typical LCS/LCSD Summary                 |
| 17.1.9.  | Figure 9  | Typical MS/MSD Summary                   |
| 17.1.10. | Figure 10 | Typical Case Narrative                   |

**17.2. Appendices**

- |         |            |                                       |
|---------|------------|---------------------------------------|
| 17.2.1. | Appendix 1 | Summary of Quality Control Procedures |
|---------|------------|---------------------------------------|

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17.2.2. Appendix 2 Demonstration of Capability

**17.3 Forms**

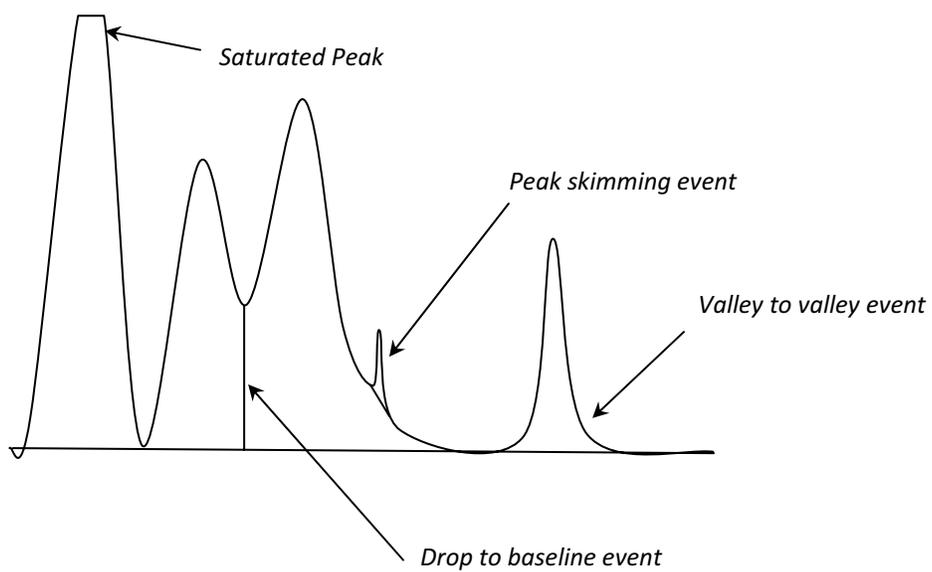
17.3.1 8015GFS Sample Preparation Log

17.3.1 8015GFA Analytical Run Log

17.3.2 8015GFM Instrument Maintenance Log

**Figure 1:** **TYPICAL PEAK EVALUATION**

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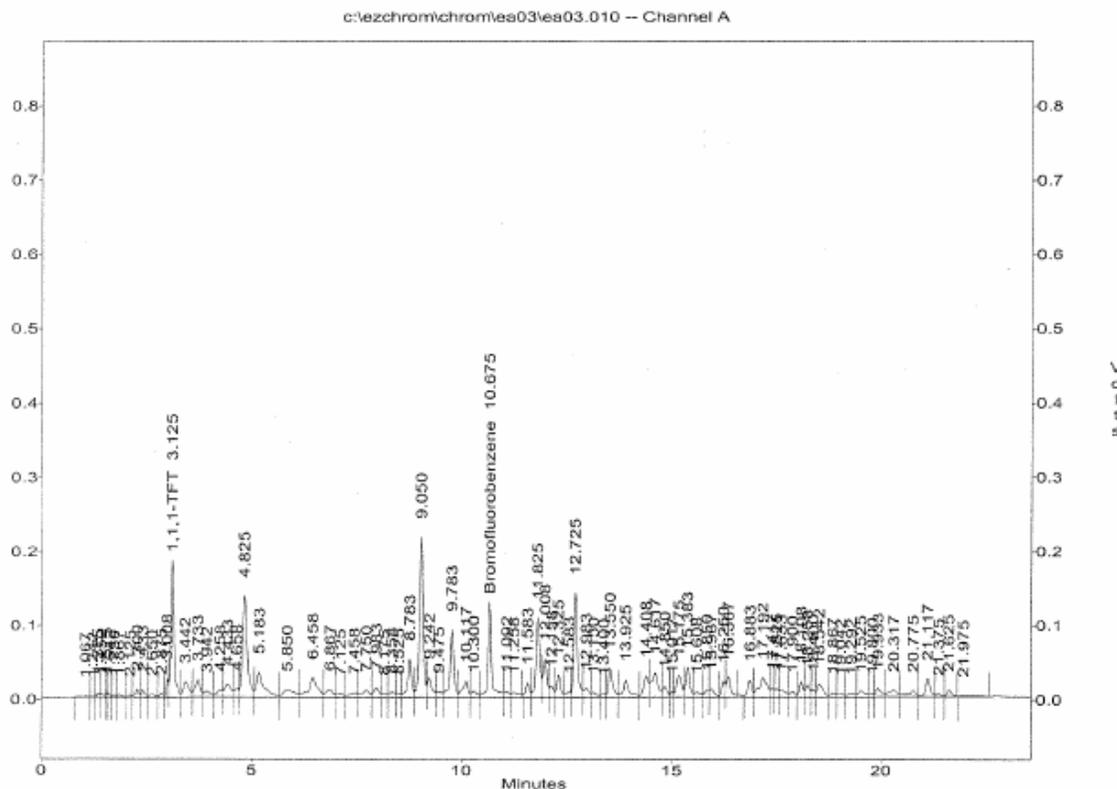
**Figure 2: TYPICAL CHROMATOGRAM**

EPA METHOD 8015 by FID  
 EMAx Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.010  
 Method : c:\ezchrom\methods\vg39a03.met  
 Sample ID : IVG39A0302 500/40  
 Acquired : Jan 03, 2014 21:37:01  
 Printed : Jan 06, 2014 12:19:37  
 User : SERGIO

Channel A Results

#	Peak Name	Ret. Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
15	1,1,1-TFT	3.125	984381.0	22722.0	43.32
41	Bromofluorobenzene	10.675	788502.0	17848.0	44.18
G1	GASOLINE (TOTAL)		14063293.0	27233.2	516.40
G2	GRO (C6-C10)		9336308.0	20606.5	453.08
G3	GRO (2MP-124TMB)		9284983.0	20571.2	451.36
G4	GRO (C5-C12)		13063346.0	26615.6	490.81
G5	GRO (C6-C12)		13013487.0	26585.6	489.49
G6	GRO (C5-C10)		9386167.0	20636.5	454.83



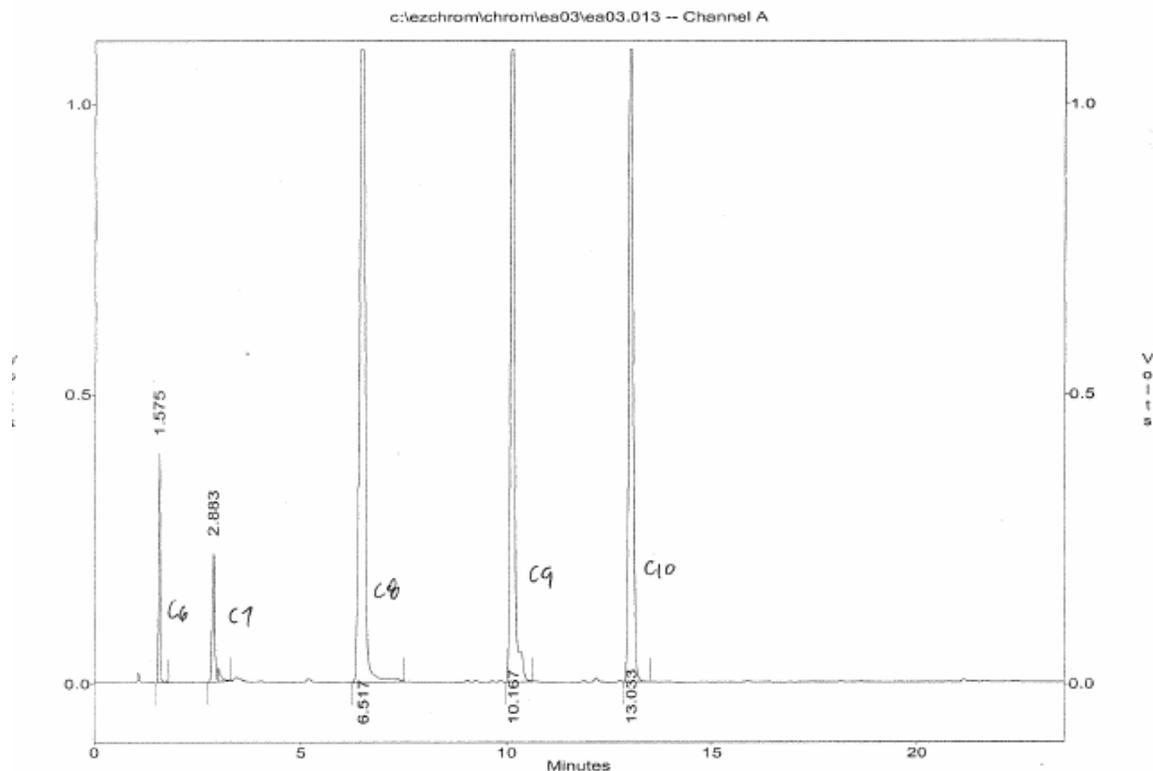
**Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS**

EPA METHOD 8015 by FID  
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.013  
 Method : c:\ezchrom\methods\vg39a03.met  
 Sample ID : GRO 1UL  
 Acquired : Jan 03, 2014 23:32:32  
 Printed : Jan 06, 2014 12:22:28  
 User : SERGIO

Channel A Results

#	Peak Name	Ret.Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE (TOTAL)		29925396.0	27233.2	1098.86
G2	GRO (C6-C10)		21311152.0	20606.5	1034.20
G3	GRO (2MP-124TMB)		21311152.0	20571.2	1035.97
G4	GRO (C5-C12)		29925396.0	26615.6	1124.35
G5	GRO (C6-C12)		29925396.0	26585.6	1125.62
G6	GRO (C5-C10)		21311152.0	20636.5	1032.69



**Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)**

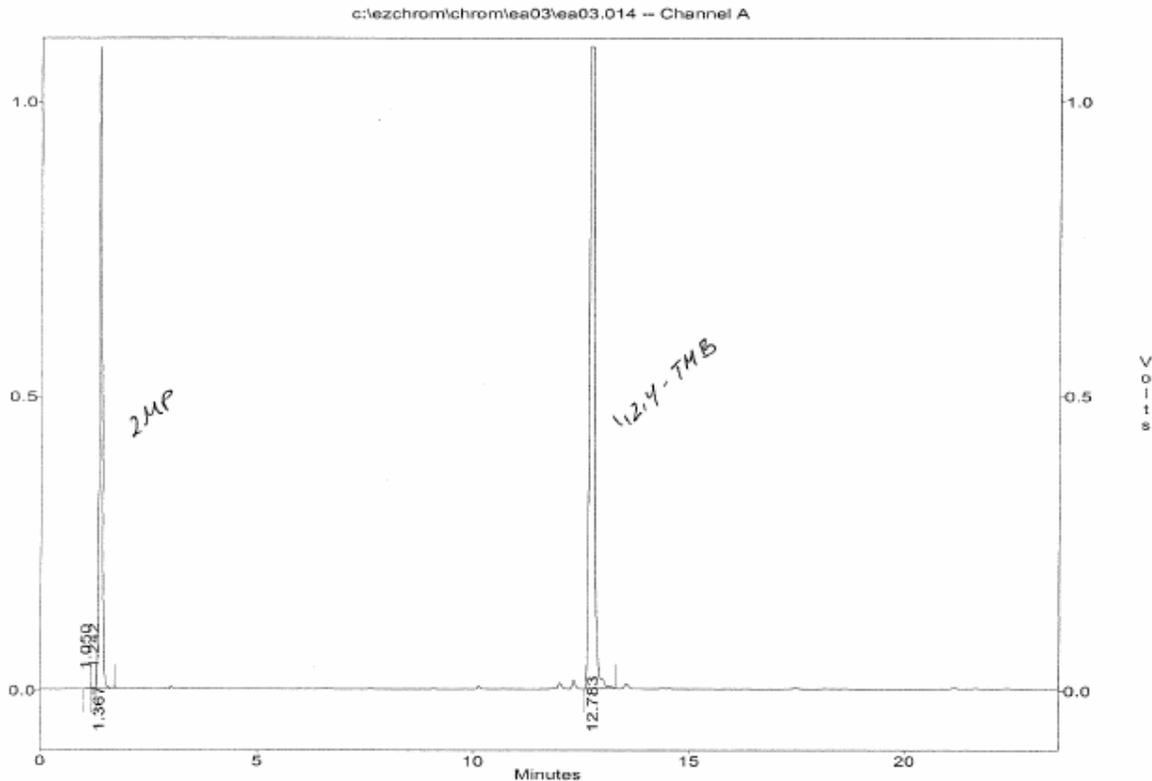
Page 1 of 1

EPA METHOD 8015 by FID  
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.014  
 Method : c:\ezchrom\methods\vg39a03.met  
 Sample ID : 2MP/1,2,4-TMB  
 Acquired : Jan 04, 2014 00:11:01  
 Printed : Jan 06, 2014 12:22:38  
 User : SERGIO

Channel A Results

#	Peak Name	Ret. Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE (TOTAL)		14307337.0	27233.2	525.36
G2	GRO (C6-C10)		9312826.0	20606.5	451.94
G3	GRO (2MP-124TMB)		0.0	20571.2	0.00
G4	GRO (C5-C12)		14304282.0	26615.6	537.44
G5	GRO (C6-C12)		9312826.0	26585.6	350.30
G6	GRO (C5-C10)		14304282.0	20636.5	693.15



**Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)**

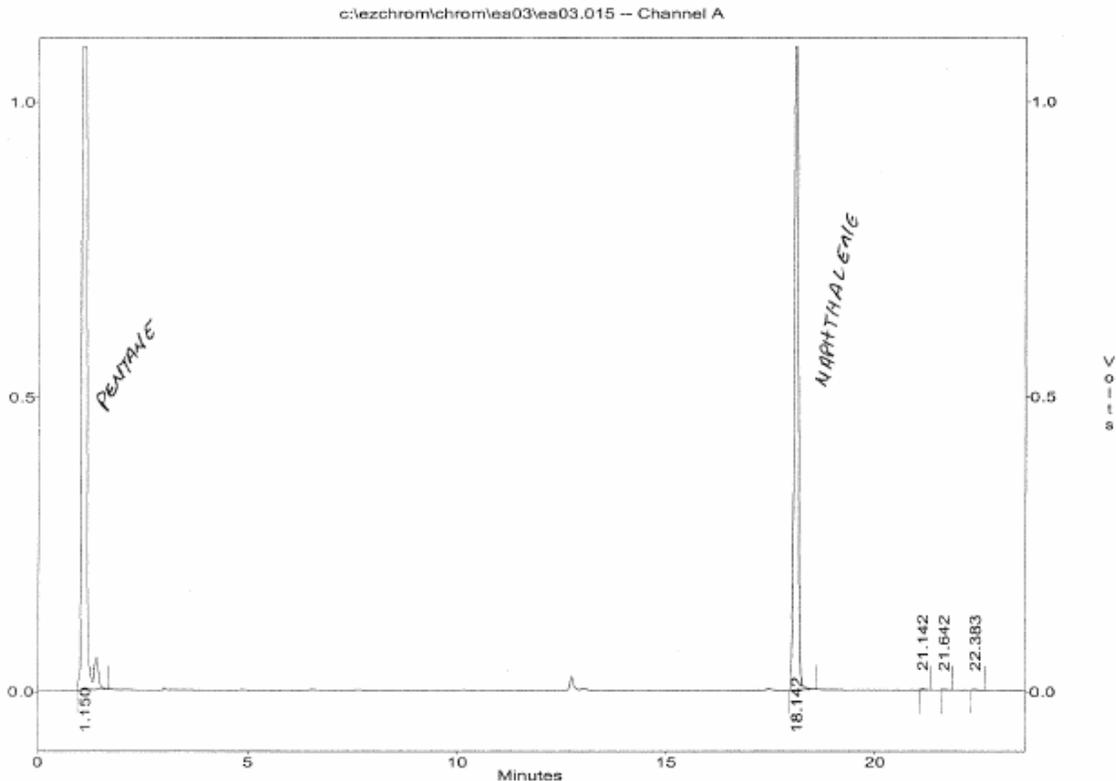
Page 1 of 1

EPA METHOD 8015 by FID  
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.015  
 Method : c:\ezchrom\methods\vg39a03.met  
 Sample ID : PENTANE/NAPHTHALENE  
 Acquired : Jan 04, 2014 00:49:28  
 Printed : Jan 06, 2014 12:22:47  
 User : SERGIO

Channel A Results

#	Peak Name	Ret.Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE (TOTAL)		17371524.0	27233.2	637.88
G2	GRO (C6-C10)		0.0	20606.5	0.00
G3	GRO (2MP-124TMB)		0.0	20571.2	0.00
G4	GRO (C5-C12)		17347616.0	26615.6	651.78
G5	GRO (C6-C12)		7450149.0	26585.6	280.23
G6	GRO (C5-C10)		9897466.0	20636.5	479.61



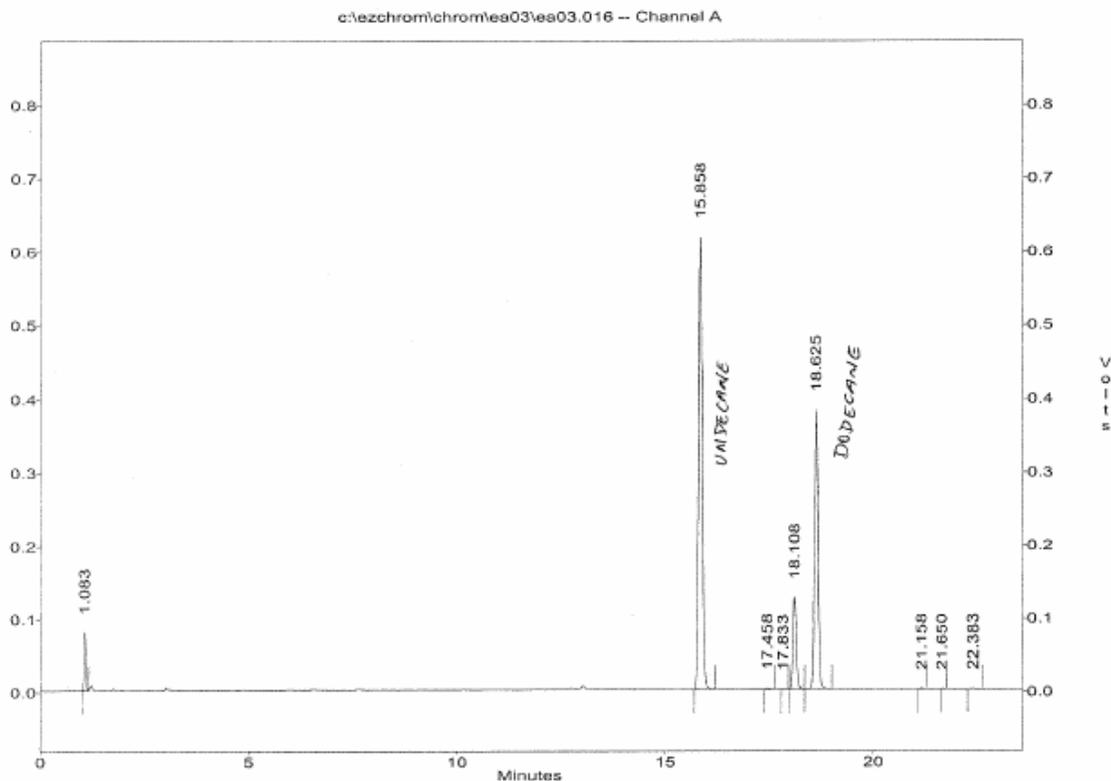
**Figure 3: TYPICAL HYDROCARBON MARKER CHROMATOGRAMS (continuation)**

EPA METHOD 8015 by FID  
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea03\ea03.016  
 Method : c:\ezchrom\methods\vg39a03.met  
 Sample ID : UNDECANE/DODECANE  
 Acquired : Jan 04, 2014 01:27:55  
 Printed : Jan 06, 2014 12:23:00  
 User : SERGIO

Channel A Results

#	Peak Name	Ret. Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
--	1,1,1-TFT	3.150	0.0	0.0	0.00
--	Bromofluorobenzene	10.700	0.0	0.0	0.00
G1	GASOLINE (TOTAL)		7098382.0	27233.2	260.65
G2	GRO (C6-C10)		0.0	20606.5	0.00
G3	GRO (2MP-124TMB)		0.0	20571.2	0.00
G4	GRO (C5-C12)		6837192.0	26615.6	256.89
G5	GRO (C6-C12)		6837192.0	26585.6	257.18
G6	GRO (C5-C10)		0.0	20636.5	0.00



**Figure 4: TYPICAL ICAL SUMMARY**

INITIAL CALIBRATION  
5030B/M8015

Lab Name : EMAX Inc  
Instrument ID : GCT39  
GC Column : DB-5  
Column size ID : 30MX.53MM  
LFID & Datetime: EA03003A 01/03/14 17:08  
LFID & Datetime: EA03004A 01/03/14 17:46  
LFID & Datetime: EA03005A 01/03/14 18:25  
LFID & Datetime: EA03006A 01/03/14 19:03  
LFID & Datetime: EA03007A 01/03/14 19:41  
LFID & Datetime: EA03008A 01/03/14 20:20  
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS						(AREA)/UNIT		MEAN	%RSD
		1.00X	2.50X	5.00X	25.00X	50.00X	75.00X				
Gasoline(TOTAL)	20.00	24652	27578	27219	29767	26888	27295	27233.2	6.0		
GRO(C6-C10)	20.00	18461	21683	20770	21960	20286	20480	20606.5	6.0		
GRO(2MP-124TMB)	20.00	18461	21683	20770	21865	20227	20422	20571.2	6.0		
GRO(C5-C12)	20.00	23119	27187	26894	29198	26465	26832	26615.6	7.4		
GRO(C6-C12)	20.00	23022	27147	26869	29193	26457	26826	26585.6	7.5		
GRO(C5-C10)	20.00	18558	21723	20794	21964	20294	20486	20636.5	5.9		
SURROGATE	X	1.00X	2.00X	3.00X	4.00X	5.00X	6.00X	MEAN	%RSD		
Bromofluorobenzene	10.00	15758	16495	16129	20469	18793	19444	17848.0	11.1		
1,1,1-Trifluorotoluene	10.00	22945	22299	21809	22278	23050	23951	22722.0	3.3		

VG39A03.MET

INITIAL CALIBRATION  
5030B/M8015

Lab Name : EMAX Inc  
Instrument ID : GCT39  
GC Column : DB-5  
Column size ID : 30MX.53MM  
LFID & Datetime: EA03003A 01/03/14 17:08  
LFID & Datetime: EA03004A 01/03/14 17:46  
LFID & Datetime: EA03005A 01/03/14 18:25  
LFID & Datetime: EA03006A 01/03/14 19:03  
LFID & Datetime: EA03007A 01/03/14 19:41  
LFID & Datetime: EA03008A 01/03/14 20:20

COMPOUND	RT OF STANDARDS (MIN)						MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.5X	5.0X	25.0X	50.0X	75.0X		FROM	TO	
Gasoline(TOTAL)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C6-C10)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(2MP-124TMB)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C5-C12)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C6-C12)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
GRO(C5-C10)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
SURROGATE	1.0X	2.0X	3.0X	4.0X	5.0X	6.0X	RT	FROM	TO	WIDTH
Bromofluorobenzene	10.700	10.683	10.683	10.675	10.675	10.675	10.682	10.639	10.725	0.043
1,1,1-Trifluorotoluene	3.192	3.158	3.167	3.117	3.125	3.125	3.147	3.023	3.271	0.124

VG39A03.MET

**Figure 5: TYPICAL CONTINUING CALIBRATION SUMMARY**

CONTINUE CALIBRATION  
 5030B/M8015

Lab Name : EMAX Inc  
 Instrument ID : GCT39  
 GC Column : DB-5  
 Column size ID : 30MX.53MM  
 Mid Conc Init LFID & Datetime: EA03006A 01/03/2014 19:03  
 Conc Cont LFID & Datetime: EA09003A 01/09/2014 13:25  
 CONC UNIT : ppb

COMPOUND	RT MINUTES	RT WINDOW		TRUE CONC	AVERAGE CF	RESULT			QL	%D LIMITS
		FROM	TO			AREA	CONC	%D		
Gasoline(TOTAL)	NA	NA	NA	500.0	27233.2	13446054	493.74	-1		20
GRO(C6-C10)	NA	NA	NA	500.0	20606.5	10381184	503.78	1		20
GRO(2MP-124TMB)	NA	NA	NA	500.0	20571.2	10231844	497.39	-1		20
GRO(C5-C12)	NA	NA	NA	500.0	26615.6	13343700	501.35	0		20
GRO(C6-C12)	NA	NA	NA	500.0	26585.6	13342070	501.85	0		20
GRO(C5-C10)	NA	NA	NA	500.0	20636.5	10382814	503.13	1		20
SURROGATE	MINUTES	FROM	TO	TRUECON	CF	AREA	CONC	%D	QL	LIMITS
Bromofluorobenzene	10.700	10.657	10.743	40.0	17848.0	731282	40.97	2		20
1,1,1-Trifluorotoluene	3.192	3.068	3.316	40.0	22722.0	921311	40.55	1		20

### Figure 6: TYPICAL RAW DATA

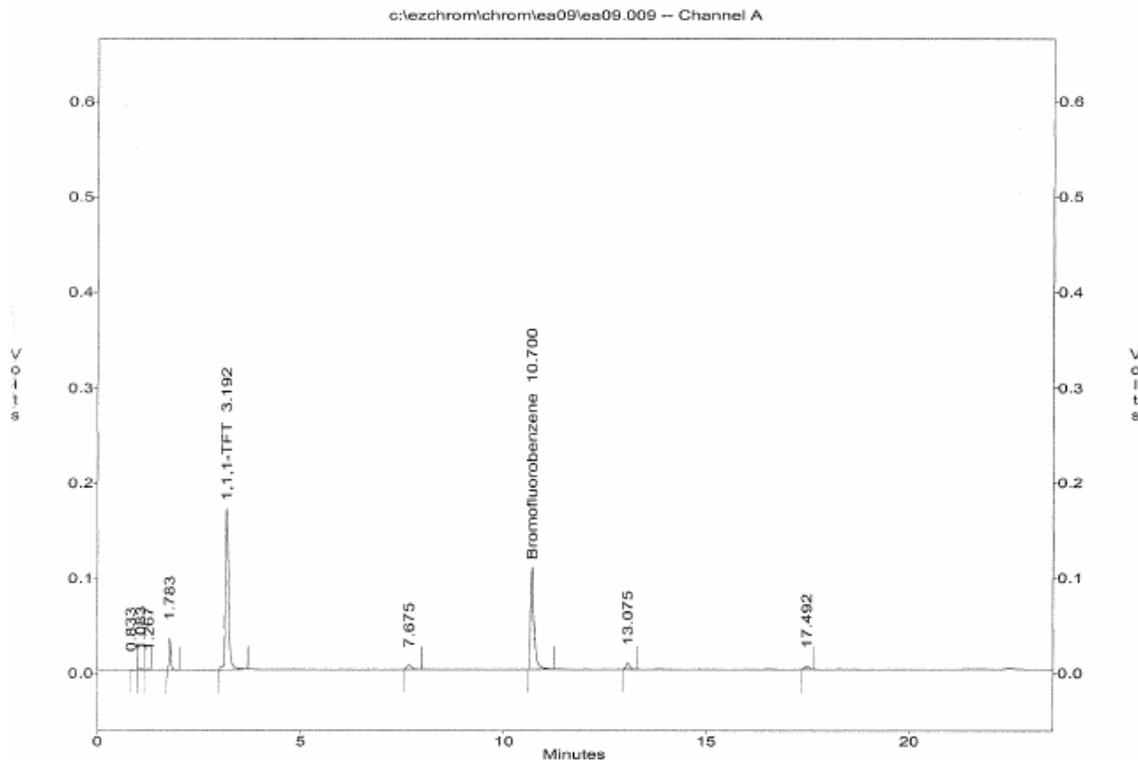
Page 1 of 1

EPA METHOD 8015 by FID  
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ea09\ea09.009  
Method : c:\ezchrom\methods\vg39a03.met  
Sample ID : 14A018-03 5.0ML W  
Acquired : Jan 09, 2014 17:21:15  
Printed : Jan 24, 2014 11:58:54  
User : SERGIO

#### Channel A Results

#	Peak Name	Ret. Time (Min)	Area	Ave. CF	ESTD Conc. (PPB)
5	1,1,1-TFT	3.192	908699.0	22722.0	39.99
7	Bromofluorobenzene	10.700	638363.0	17848.0	35.77
G1	GASOLINE (TOTAL)		221859.0	27233.2	8.15
G2	GRO (C6-C10)		137610.0	20606.5	6.68
G3	GRO (2MP-124TMB)		137610.0	20571.2	6.69
G4	GRO (C5-C12)		212863.0	26615.6	8.00
G5	GRO (C6-C12)		209157.0	26585.6	7.87
G6	GRO (C5-C10)		141316.0	20636.5	6.85



**Figure 7: TYPICAL SAMPLE RESULT SUMMARY**

METHOD 5030B/8015C  
 TOTAL PETROLEUM HYDROCARBONS BY PURGE AND TRAP

```

=====
Client       : XYZ, INC.                Date Collected: 01/07/14
Project      : CLEAN WATER             Date Received: 01/08/14
Batch No.    : 14A018                  Date Extracted: 01/09/14 17:21
Sample ID    : A5-MW15-010714         Date Analyzed: 01/09/14 17:21
Lab Samp ID  : A018-03                 Dilution Factor: 1
Lab File ID  : EA09009A               Matrix          : WATER
Ext Btch ID  : VG39A06                % Moisture     : NA
Calib. Ref.  : EA09003A               Instrument ID   : GCT039
=====
  
```

PARAMETERS	RESULTS (mg/L)	LOQ (mg/L)	DL (mg/L)	LOD (mg/L)
GASOLINE	0.0067J	0.020	0.0050	0.010
SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
BROMOFLUOROBENZENE	0.0358	0.04000	89.4	60-140

Parameter      H-C Range  
 Gasoline        C6-C10

Figure 8:

TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER  
 BATCH NO.: 14A018  
 METHOD: METHOD 5030B/8015C

=====

MATRIX: WATER % MOISTURE: NA  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: MBLK1W  
 LAB SAMP ID: VG39A06B VG39A06L VG39A06C  
 LAB FILE ID: EA09006A EA09004A EA09005A  
 DATE EXTRACTED: 01/09/1415:23 01/09/1414:03 01/09/1414:42 DATE COLLECTED: NA  
 DATE ANALYZED: 01/09/1415:23 01/09/1414:03 01/09/1414:42 DATE RECEIVED: 01/09/14  
 PREP. BATCH: VG39A06 VG39A06 VG39A06  
 CALIB. REF: EA09003A EA09003A EA09003A

ACCESSION:

PARAMETER	BLNK RSLT (mg/L)	SPIKE AMT (mg/L)	BS RSLT (mg/L)	BS % REC	SPIKE AMT (mg/L)	BSD RSLT (mg/L)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Gasoline	ND	0.500	0.477	95	0.500	0.459	92	4	60-130	30

=====

SURROGATE PARAMETER	SPIKE AMT (mg/L)	BS RSLT (mg/L)	BS % REC	SPIKE AMT (mg/L)	BSD RSLT (mg/L)	BSD % REC	QC LIMIT ( % )
Bromofluorobenzene	0.0400	0.0395	99	0.0400	0.0390	97	70-130

Figure 9:

TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN WATER  
 BATCH NO.: 14A018  
 METHOD: METHOD 5030B/8015C

MATRIX: WATER % MOISTURE: NA  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: A5-MW15-010714  
 LAB SAMP ID: A018-03 A018-03M A018-03S  
 LAB FILE ID: EA09009A EA09010A EA09011A  
 DATE EXTRACTED: 01/09/1417:21 01/09/1417:59 01/09/1418:38 DATE COLLECTED: 01/07/14  
 DATE ANALYZED: 01/09/1417:21 01/09/1417:59 01/09/1418:38 DATE RECEIVED: 01/08/14  
 PREP. BATCH: VG39A06 VG39A06 VG39A06  
 CALIB. REF: EA09003A EA09003A EA09003A

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	SPIKE AMT (mg/L)	MS RSLT (mg/L)	MS % REC	SPIKE AMT (mg/L)	MSD RSLT (mg/L)	MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Gasoline	0.006683	0.500	0.445	88	0.500	0.465	92	4	50-130	30

SURROGATE PARAMETER	SPIKE AMT (mg/L)	MS RSLT (mg/L)	MS % REC	SPIKE AMT (mg/L)	MSD RSLT (mg/L)	MSD % REC	QC LIMIT ( % )
Bromofluorobenzene	0.0400	0.0390	98	0.0400	0.0392	98	60-140

Figure 10:

TYPICAL CASE NARRATIVE

---

CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN WATER  
SDG : 14A018

METHOD 5030B/8015C  
TOTAL PETROLEUM HYDROCARBONS BY PURGE AND TRAP

A total of ten (10) water samples were received on 01/08/14 for TPH Gasoline analysis, Method 5030B/8015C in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for VG39A06L/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the samples in this SDG. Percent recoveries for A018-03M/S were within project QC limits.

Surrogate

Surrogate was added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter.

**Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES**

QC Procedure	Frequency	Acceptance Criteria	Corrective Action	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Five-point initial calibration for all analytes	Initially; as needed	ACF RSD: ≤ 20%	Correct the problem then repeat initial calibration		
Initial calibration verification (ICV)	After initial calibration	Within ± 20% of expected value	Correct the problem then repeat initial calibration		
Calibration verification (DCC)	Every 12 hours of analysis time and at the end of analysis sequence	Within ± 20% of expected value	Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification		
Method blank	One per preparation batch	No analytes detected ≥ ½ LOQ	If sample results are ND or contamination is < 10X of sample result, consult with the PM if results are reportable. Otherwise, determine the source of contamination and correct the problem. Reanalyze method blank and all samples processed with the contaminated blank. If re-analysis is not possible, apply B to specific analyte(s) on all associated.		
LCS	One LCS per preparation batch	Within project QC Limits	Re-prep and reanalyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard, and method blank	Within project QC Limits	If no apparent matrix interference is observed, reanalyze the sample. Otherwise inform the PM for further instruction.		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Refer to project QC Limits	Ensure that spike concentration, spike addition was accurate and calculation is correct. If chromatogram exhibits matrix interference narrate observation in the case narrative.		
Chromatogram	All sample results	Within calibration range  No saturated peak(s)	Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range.		
Notes: Discrete peaks are included for GRO Discrete peaks are subtracted for Gasoline Refer to PSR for flagging criteria. Report results between LOD and LOQ.				Reviewed By	
				Date	

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
GASOLINE RANGE ORGANICS  
METHOD: SW 8015C**

SOP: EMAX-8015G  
Conc Unit: µg/L  
Sample Amount(ml): 5

Instrument ID: 39  
Analysis date: 1/6/2014  
Analyzed by: Cervantes, Sergio

PARAMETER	EA06004A	EA06005A	EA06006A	EA06007A	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	VG39A01L	VG39A01C	VG39A02L	VG39A02C							
Gasoline(TOTAL)	458	485	447	426	500	454	91	24.5	5	70 - 130	PASSED
GRO(C6-C10)	503	540	501	477	500	505	101	25.8	5	70 - 130	PASSED
GRO(2MP-124TMB)	504	542	503	480	500	507	101	25.4	5	70 - 130	PASSED
GRO(C5-C12)	461	490	453	432	500	459	92	24.2	5	70 - 130	PASSED
Bromofluorobenzene	42.3	42.8	41.7	39.4	40	41.5	104	1.50	4	70 - 130	PASSED
1,1,1-Trifluorotoluene	46.4	46.3	45.6	45.6	40	46.0	115	0.43	1	70 - 130	PASSED

SOP: EMAX-8015G  
Conc Unit: µg/Kg  
Sample Amount(g): 5

Instrument ID: 39  
Analysis date: 1/6/2012  
Analyzed by: Cervantes, Sergio

PARAMETER	EA06013A	EA06014A	EA06015A	EA06016A	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	GMA001SL	GMA001SC	GMA001SX	GMA001SY							
Gasoline(TOTAL)	20480	19683	20590	20124	25000	20200	81	409	2	60 - 130	PASSED
GRO(C6-C10)	22739	21724	22671	22202	25000	22300	89	472	2	60 - 130	PASSED
GRO(2MP-124TMB)	22676	21739	22618	22216	25000	22300	89	433	2	60 - 130	PASSED
GRO(C5-C12)	20651	19816	20681	20251	25000	20300	81	406	2	60 - 130	PASSED
Bromofluorobenzene	2228	2109	2176	2166	2000	2170	109	49	2	70 - 130	PASSED
1,1,1-Trifluorotoluene	2337	2246	2335	2338	2000	2310	116	45	2	70 - 130	PASSED

8015GFS:

SAMPLE PREPARATION LOG



EXTRACTION LOG  
*for*  
 PURGEABLE TPH

Page 1

**Note:** For samples, relevant QCs/Standards extracted,  
 refer to attached extraction sequence.

Book #.: E39-036

Preparation Batch:

**Comments:**

Matrix

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

SOP	Rev. #
<input type="checkbox"/> EMAX-5035	3
<input type="checkbox"/> EMAX-AK101	2
<input type="checkbox"/> EMAX-	

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Standards	ID	Amount Added (ml)
Surrogate		
LCS/MS		
Methanol		
Methanol w/ Surrogate		

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Reagent	Lot # / ID
Silica Sand	

Prepared By: \_\_\_\_\_

Prepared By: \_\_\_\_\_

Standard Added By: \_\_\_\_\_

Standard Added By: \_\_\_\_\_ Checked By: \_\_\_\_\_

Extract Location: \_\_\_\_\_

Extract Location: \_\_\_\_\_

Disposal By: \_\_\_\_\_

Disposal By: \_\_\_\_\_ Disposal Date: \_\_\_\_\_





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SOP REVIEW FORM

EMAX-8081  
SOP No.

Rev. 8  
Revision Number

ORGANOCHLORINE PESTICIDES BY GC  
Title

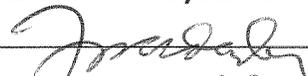
Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Data Reduction	/			
- Calculations	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

No revision needed.

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

  
FARINA MADAMBA

Date:

6/4/15

## STANDARD OPERATING PROCEDURES

**ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY**

SOP No.: EMAX-8081 Revision No. 8 Effective Date: 16-Jun-14

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Laboratory Director

Control Number: 8081-08-

**1.0 SCOPE AND APPLICATION**

- 1.1. This procedure is used to determine the concentration of various Organochlorine Pesticides in soil, sediment, sludge, and wastewater samples by gas chromatography method. This SOP is an adaptation of EPA 8081B. Since EPA 8081B is an update and enhancement of EPA 8081A, this SOP is also applicable to EPA 8081A.

**2.0 SUMMARY OF METHOD**

- 2.1. This method provides gas chromatographic conditions for the identification and quantitation of Organochlorine Pesticides with dual Electron Capture Detectors (ECD). The samples are extracted in methylene chloride and exchanged to hexane before GC analysis.
- 2.2. **Interferences**
- 2.2.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. Avoiding contact with any plastic materials and checking all solvents for phthalate contamination can minimize interferences. Glassware must be scrupulously cleaned.
- 2.2.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is most likely present in sediment samples. The TBA procedure, GPC or other cleanup technique can be used for sulfur removal.
- 2.2.3. Other interferences such as aliphatic compounds, aromatics and nitrogen-containing compounds may be eliminated by using Florisil cleanup.

**3.0 DETECTION LIMITS****3.1. Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Table 10 lists the DL, LOD and LOQ values of the target analytes of this method.

**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.

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- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered “under-range”. A lower dilution factor is required for proper quantitation.
- 4.3. The linear dynamic range for this method as determined in this SOP is listed on the table below.

Analytes	Water (µg/L)	Soil (µg/Kg)
alpha-BHC, Endosulfan I, gamma-BHC, Heptachlor, Aldrin, alpha-Chlordane, beta-BHC, delta-BHC, gamma-Chlordane, Heptachlor Epoxide	0.1 – 0.8	2 – 26
4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endrin, Endosulfan II, Endosulfan Sulfate, Endrin Aldehyde, Endrin Ketone	0.1 – 1.6	2 – 52
Methoxychlor	1 – 8	10– 260
Toxaphene	2 – 20	50 – 660
Technical Chlordane	1 – 15	50 – 500
2,4'-DDD, 2,4'-DDE, 2,4'-DDT, Oxychlordane, cis-Nonachlor, trans-Nonachlor, Mirex	0.1 – 0.8	2 – 26

**5.0 SAMPLE HOLDING TIME AND PRESERVATION****5.1. Holding Time**

- 5.1.1. Extract water and soil samples within 7 and 14 days from date of collection, respectively.
- 5.1.2. Analyze extracts within 40 days after extraction completion date.

**5.2. Preservation**

- 5.2.1. Store samples and extract at  $\leq 6^{\circ}\text{C}$ .

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA04 Detection Limit Study
- 6.3. EMAX-QA08 Corrective Action
- 6.4. EMAX-QC01 Quality Control for Chemicals
- 6.5. EMAX-QC02 Analytical Standard Preparation
- 6.6. EMAX-QC07 Glassware Cleaning
- 6.7. EMAX-SM03 Waste Disposal
- 6.8. EMAX-SM04 Analytical and QC Sample Labeling
- 6.9. EMAX-3520 Extraction, Continuous Liquid/Liquid
- 6.10. EMAX-3550 Extraction, Pulse Sonication
- 6.11. EMAX-3540 Extraction, Soxhlet
- 6.12. EMAX-3620 Cleanup, Florisil

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6.13. EMAX-3660 Cleanup, Sulfur

**7.0 SAFETY**

- 7.1. Read all SDS of chemicals listed in this SOP.
- 7.2. ECD contains minute quantity of Radioactive Ni (63). Conduct a wipe test (experienced personnel or manufacturer only) semi-annually or sooner if potential problem is suspected.
- 7.3. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure.
- 7.4. If, for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amounts of tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS, AND REAGENTS****8.1. Instruments and Supplies**

Gas Chromatograph	PE Clarus 680
Detector	Dual Electron Capture Detectors
Column	RTX CLPEST I (30 m x 0.32 mm x 0.32 $\mu$ m) RTX CLPEST II (30 m x 0.32 mm x 0.25 $\mu$ m) (Alternate columns may be used after verification of performance)
Data System	EZ Chrom Elite
Auto Sampler	PE Clarus 680 or equivalent
Gas	ultra-high purity nitrogen Peak Scientific Hydrogen Generator PH-600 PE Hydrogen Generator PGX Series 3
Microsyringes	10, 25, 100 and 500 $\mu$ l with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes
Transfer Pipette	Pasteur

**8.2. Chemicals and Reagents**

Solvent [GC-grade]	Methylene Chloride, Hexane, Acetone
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**9.0 STANDARDS****9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX-SM04, respectively.

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9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

**9.2. Stock Standard**

9.2.1. Purchase Primary Calibration stock standards as certified solutions in one mixture. After opening, transfer the stock standard to an inert amber vial and store with a minimum of headspace.

9.2.2. Purchase a Secondary set of stock standards from a different source to verify the concentration of the first set of standard. Treat the secondary standard similarly as the primary standard.

9.2.3. Purchase LCS/MS, surrogate and performance evaluation standards as certified solutions from various suppliers.

9.2.4. All standards shall be stored at  $\leq 6^{\circ}\text{C}$ .

**9.3. Intermediate Standard**

9.3.1. Prepare intermediate standards as suggested in Table 3 (primary source) and Table 4 (secondary source).

9.3.2. Store all prepared standards in an inert vial with minimum headspace at  $\leq 6^{\circ}\text{C}$ .

**9.4. Initial Calibration Standard (ICAL)**

9.4.1. Prepare five or more calibration standards as suggested in Table 2 from primary intermediate standard (refer to Table 3).

**9.5. Initial Calibration Verification (ICV)**

9.5.1. Prepare ICV at concentration levels suggested in Table 5 using intermediate standard from second source stock standard (refer to Table 4).

**9.6. Daily Calibration Check Standard (DCC)**

9.6.1. Prepare DCC from the same source as the ICAL standard as suggested in Table 5.

**9.7. Surrogate Standard**

9.7.1. Prepare surrogate standard as suggested in Table 6.

**9.8. LCS/MS Spike Standard**

9.8.1. Prepare LCS solution as suggested in Table 4.

9.8.2. Prepare MS spike standard as suggested in Table 7.

**9.9. Performance Evaluation Mixture (PEM)**

9.9.1. Prepare PEM as suggested in Table 8.

**10.0 PROCEDURES****10.1. Sample Preparation**

10.1.1. Prepare aqueous samples as described in EMAX-3520 or EMAX-3510.

10.1.2. Prepare solid samples as described in EMAX-3550, EMAX-3540 or EMAX-3545.

10.1.3. Perform extract clean up (if necessary) as described in EMAX-3620, EMAX-3640 or EMAX-3660, whichever is appropriate.

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10.2.1. Method 8081A requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 1 to 5  $\mu$ l.

## 10.2.2. Gas Pressure

N<sub>2</sub> gas line : 60 – 90 psi

H<sub>2</sub> gas line : 60 – 90 psi

10.2.3. Detector Temperature : 305°C (Inst. E8 and F9)

10.2.4. Injector Temperature : 220°C (Inst. E8 & F9)

10.2.5. Make-up Gas Flow : N<sub>2</sub> at 40 mL/min

## 10.2.6. Carrier Gas Flow

For Temperature Program (Inst. F9) : 3.8 mL/min

For Temperature Program (Inst. E8) : See flow program below

Carrier Gas Ramp	Rate (mL/min)	Setpoint (mL/min)	Hold (min)
Initial	0	2.7	5.5
1	2.0	4.0	20.0

## 10.2.7. Oven Temperature Program (Instrument E8)

Oven Ramp	Rate (°C/min)	Temp (°C)	Hold (min)
Initial	0	140	0.50
1	25	230	2.00
2	5	260	0.20
3	25	290	2.50

## 10.2.8. Oven Temperature Program (Instrument F9)

Oven Ramp	Rate (°C/min)	Temp (°C)	Hold (min)
Initial	0	130	0.50
1	25	230	2.00
2	5	260	0.20
3	25	290	3.00

**10.3. Calibration****10.3.1. Performance Evaluation Check**

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- 10.3.1.1. Analyze instrument blank and a PEM containing DDT and Endrin to monitor the system performance at 12-hour interval prior to performing any calibration.
- 10.3.1.2. Calculate the breakdown by using Equations 10.6.6 (%B<sub>T</sub>) for DDT and 10.6.7 (%B<sub>E</sub>) for Endrin.
- 10.3.1.3. Check Appendix 1 for acceptance criteria before proceeding with sample analysis.
- 10.3.1.4. If system failed to meet the acceptance criteria, refer to Section 12 for corrective action.
- 10.3.2. **Initial Calibration (ICAL)**
- 10.3.2.1. Perform ICAL if instrument is new, ICV or DCC failed to meet acceptance criteria or after a major instrument repair.
- 10.3.2.2. A minimum of five calibration standards, or as suggested in Table 1, over the concentration range of interest are sequentially injected into the GC. Refer to Table 1 for ICAL concentrations. Peak areas are obtained from each analyte.
- 10.3.2.3. Application of ICAL Curve for Quantitation
- 10.3.2.3.1. Generate a summary of calibration factors for each analyte at each concentration using Eq-10.6.1. Calculate the Average Calibration Factor (ACF), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to, Eq. 10.6.2, Eq. 10.6.3 and Eq. 10.6.4, respectively.
- If RSD is  $\leq 20\%$  ACF may be applied.
  - Apply Inverse Weighting Factor ( $1/y$  or  $1/y^2$ ;  $y$  being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.995$ .
  - Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.995$ .
  - It may be appropriate to force the regression through zero for specific analytes<sup>1</sup>. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.
- 10.3.2.4. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.
- 10.3.2.5. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.
- 10.3.3. **Initial Calibration Verification (ICV)**
- 10.3.3.1. Analyze ICV prepared from another source as described in Section 9.5 to verify the concentrations of the ICAL.

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<sup>1</sup> SW846 Method 8000B, Section 7.5.3; SW846 Method 8000C, Section 11.5.2.1

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10.3.3.2. Calculate the CF and the percent difference (%D) according to Eq-10.6.1 and 10.6.5 respectively. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

**10.3.4. Multicomponent Target Analyses**

10.3.4.1. For multicomponent target analytes (Toxaphene), a five-point calibration standard shall be included in initial calibration for pattern recognition and quantitation.

10.3.4.2. Integrate the total response of the chromatogram to obtain the total area. Calculate the calibration factor (CF) by using Equation 10.6.1.

**10.3.5. Daily Calibration Check (DCC)**

10.3.5.1. Analyze DCC at the start of the 12-hour shift prior to sample analysis and close the analytical run with an ending DCC.

10.3.5.2. Calculate the %D by using Equation 10.6.5. Check Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

**10.4. Analysis****10.4.1. Analytical Sequence**

10.4.1.1. Following the instrument data acquisition software, prepare the analytical sequence file as suggested below:

- Pesticide Prime – 20/200 ppb pesticide injected at the beginning of analytical sequence if the GC has not been used for a day or more
- IB – instrument blank
- PEM – performance evaluation mixture
- ICAL – initial calibration standards
- ICV or DCC1 – initial calibration verification or continuing calibration standard
- MB – method blank
- LCS – lab control sample
- Samples – up to 12 hours
- DCC2 – continuing calibration standard or ending DCC

**10.4.2. Sample Analysis**

10.4.2.1. Transfer a minimum of 0.5 ml of extract to a 2-ml auto sampler vial (or equivalent) using a Pasteur pipette. Seal the vial with a polypropylene screw cap with PTFE/red silicone rubber septa. Similarly, prepare the analytical standards and QC samples.

10.4.2.2. Introduce sample extract into the GC using direct injection technique (1 to 5  $\mu$ l) after all system quality control criteria have been met.

10.4.2.3. If the response exceeds the linear range of the system, dilute the sample and re-analyze.

**10.4.3. Sample Result Evaluation**

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10.4.3.1. Check QC parameters as soon as the data is available.

- ✓ Check LCS recoveries against Appendix 1.
- ✓ Check MB that it is project compliant.
- ✓ Check retention time.
- ✓ Check surrogate recoveries against Appendix 1.
- ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, fill-up an NCR and order re-extraction for the affected sample(s).

10.4.3.2. Positive identification is made when a peak falls within the retention time window of a target analyte on both columns established by the standard reference compound.

10.4.3.3. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:

- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW (refer to table 7) whichever is greater.
- ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.

10.4.4. **Retention Time Windows**

10.4.4.1. Establishing RTW

- 10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.
- 10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte.
- 10.4.4.1.3. The width of RTW is defined by  $\pm 3X$  SD obtained from 10.4.4.1.2.

10.4.4.2. Evaluating RTW

- 10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained or use the CLP<sup>1</sup> retention time window (refer to Table 9) which ever is narrower.
- 10.4.4.2.2. For new instruments, in the interim use the CLP retention time window (refer to Table 9) until RTW is obtained for the new instrument parameters condition.

<sup>1</sup> CLP-OLM4.2 Table 1 D-79/PEST

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## 10.4.4.3. Application of RTW

10.4.4.3.1. Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.4.4.3.2. Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing the analyses.

## 10.4.4.4. Updating RTW

10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacement of detector or column; temperature program change, etc.)

10.4.5. **Manual Integration**

10.4.5.1. Refer to EMAX-DM01 for details of manual integration.

10.4.6. **Dealing with Carryover**

- ✓ Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- ✓ If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- ✓ If there was a target analyte detected as found in the sample following the sample that exceeded the calibration range (for pesticides concentration  $\geq 500/1000/5000$  ppb; for Toxaphene concentration  $\geq 25$  ppm; for chlordane concentration  $\geq 10$  ppm and for PCBs concentration  $\geq 100$  ppm) re-analyze the sample to rule-out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. **Data Reduction**

10.5.1. Check the chromatogram of positively identified peaks.

- ✓ Peaks fall within the established retention time window on both columns.
- ✓ Peaks are sharp and not saturated.
- ✓ Peaks are properly integrated (refer to Figure 1 for Peak Evaluation Techniques).
- ✓ Target analyte peak is present in both columns to confirm positive identification.

10.5.2. Positive identification is confirmed when the identified analyte is present in both columns. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Equation 10.6.10.2.

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- 10.5.2.1. If the RPD is less than 40% and the peaks do not indicate any anomalies, report the higher result.
- 10.5.2.2. If the RPD is less than 40% and one of the peaks indicate an anomaly, report the result from the better peak.
- 10.5.2.3. If the RPD is greater than 40%, use professional judgment to select the most appropriate result. If no evidence of any chromatographic interference, report the higher result.

**10.6. Calculations****10.6.1. Calculate for Calibration Factor (CF)**

$$CF = \frac{R_a}{C_k} \quad \text{Eq.-10.6.1}$$

*where:* $CF$  - is the calibration factor $R_a$  - is the analyte response measured in peak area $C_k$  - is the known concentration of the analyte in  $\mu\text{g/L}$ **10.6.2. Calculate for Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}} \quad \text{Eq.-10.6.2}$$

*where:* $SD$  - is the standard deviation $x_i$  - is the result at the  $i$ th measurement $\bar{x}$  - is the mean $N$  - is the number of measurements**10.6.3. Calculate for Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \left[ \frac{SD}{ACF} \right] 100 \quad \text{Eq.-10.6.3}$$

*where:* $\%RSD$  - is the percent relative standard deviation $SD$  - is the standard deviation $ACF$  - is the average calibration factor**10.6.4. Calculate for Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF}{N} \quad \text{Eq.-10.6.4}$$

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$$y = ax + b \quad \text{Eq.-10.5.1.3}$$

*where:* $y$  = Response factor $x$  = Concentration $a$  =  $x_1$  = slope of the line

$$a = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

*where:* $\bar{x}$  = Average of amount ratios $\bar{y}$  = Average of response ratios $b = x_0$  = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

**10.6.6. Calculate for Inverse Weighting Factor**

$$y = ax + b \quad \text{Eq.-10.5.1.4}$$

*where:* $y$  = Response Factor $x$  = Concentration $a$  =  $x_1$  = slope of the line

$$a = \frac{\sum [(x - x_a)(y - y_a)]}{\sum (x - x_a)^2}$$

*where:*

$$x_a = \sum [x(1/x)] / \sum (1/x)$$

$$y_a = \sum [y(1/x)] / \sum (1/x) \text{ or}$$

$$x_a = \sum [x(1/x^2)] / \sum (1/x^2)$$

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$$y_a = \Sigma \left[ y(1/x^2) / \Sigma (1/x^2) \right]$$

b = x0 = intercept of the line

$$b = y_a - a * x_a$$

10.6.7. Calculate for Percent Difference for DCC from ACF

$$\%D = \left[ \frac{CF - ACF}{ACF} \right] 100 \quad \text{Eq.-10.6.5}$$

where:

%D - is the % Difference

ACF - is the Average Calibration Factor

CF - is the Calibration Factor of the DCC

10.6.8. Calculate for % Breakdown for DDT (%B<sub>T</sub>).

$$\%B_T = \frac{A_D + A_E}{A_T + A_D + A_E} \quad \text{Eq.-10.6.6}$$

where:

%B<sub>T</sub> - % DDT Breakdown

A<sub>D</sub> - Total area of DDD

A<sub>E</sub> - Total area of DDE

A<sub>T</sub> - Total area of DDT

10.6.9. Calculate for % Breakdown for Endrin (%B<sub>E</sub>)

$$\%B_E = \frac{A_A + A_K}{A_E + A_A + A_K} \quad \text{Eq.-10.6.7}$$

where:

%B<sub>E</sub> - % Endrin Breakdown

A<sub>A</sub> - Total area of Endrin Aldehyde

A<sub>K</sub> - Total area of Endrin Ketone

A<sub>E</sub> - Total area of Endrin

10.6.10. Sample Results10.6.10.1. **Water Samples**

$$C = \left( \frac{R_a}{ACF} \right) \left( \frac{V_e}{S_a} \right) DF \quad \text{Eq.-10.6.8.1}$$

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$$C = \left( \frac{R_a}{ACF} \right) \left( \frac{V_e}{S_a (\% \text{ Solid})} \right) DF \quad \text{Eq.-10.6.8.2}$$

*where* $C$  - Concentration of analyte to be measured ( $\mu\text{g/kg}$ ) $R_a$  - Total response of analyte in peak area $ACF$  - Average response factor $V_e$  - Volume of extract in ml $S_a$  - Sample Amount in g $\% \text{ Solid} = \frac{100 - \% \text{ moisture}}{100}$  $DF$  - Dilution factor of the sample extract**10.6.11. Multi-peak Compound in Sample (Toxaphene)**

10.6.11.1. Total area is integrated and concentration is determined by equation 10.6.8.1 or 10.6.8.2.

**10.6.12. Accuracy and Precision****10.6.12.1. Percent Recovery**

$$\%R = \frac{C_f - C}{C_s} * 100 \quad \text{Eq.-10.6.10.1}$$

*where:* $\%R$  - percent recovery $C_f$  - concentration found in spiked sample $C$  - concentration of unspiked sample (For LCS,  $C=0$ ) $C_s$  - theoretical concentration of surrogate spike**10.6.12.2. Relative Percent Difference (RPD)**

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$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.6.10.2}$$

*where:*

%RPD - Relative Percent Difference

C1 - Measured concentration of the first sample aliquot

C2 - Measured concentration of the second sample aliquot

**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WDB1C.exe.
- 10.7.2. Generate Lab Chronicle using LABCHRN1.exe.
- 10.7.3. Generate sample results using F1NV3C.exe.
- 10.7.4. Generate the QC Summary file using QCV3CN.exe.
- 10.7.5. Generate the case narrative using CN1.exe.
- 10.7.6. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
  - Sample Results
  - LCS Summary
  - MS/MSD Summary
  - DCC Summary
  - ICAL Summary
  - ICV Summary
  - Copy of Analysis Log
  - Copy of Preparation Log

**10.8. Data Review**

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the Project Specific Requirements (PSR).
  - ✓ Check that all samples required for analysis are performed.
  - ✓ Check that samples are extracted and analyzed within Holding Time.
  - ✓ Check that all calibration requirements are fulfilled.
  - ✓ Check the chromatogram of all positively identified peak(s).
  - ✓ Check surrogate recoveries against required limits.
  - ✓ Check that concentration of target analytes are within calibration range.

If any of the above checkpoints indicate a problem, re-analysis is required.

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10.8.2. Review the case narrative and edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analysis package for secondary review.

**10.9. Preventive Maintenance**

10.9.1. Refer to Form 8081FM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

<b>Maintenance Activity</b>	<b>Description</b>	<b>Frequency</b>
Autosampler Check	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check liner as necessary. Check instrument performance (e.g., daily calibration check, instrument blank, DDT/Endrin breakdown).	Daily prior to analysis
Documentation	Record maintenance in instrument service logs.	Daily prior to analysis
Leak Test	Perform inlet pressure decay test.	Every 6 months or as necessary
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector where applicable.	Every 6 months or as necessary
Check Flow Path Components	Check and replace the following as necessary: tubing assembly, union, sample probe, and loop.	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting.	Once a year

**11.0 QUALITY CONTROL****11.1. Preparative Batch**

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD (when required by the project) and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, prepare LCS/LCD to check for precision.

## STANDARD OPERATING PROCEDURE

**ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY**

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11.1.3. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.

11.1.4. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within  $\pm 20\%$  of the expected value.

**11.2. Analytical Batch QC**

11.2.1. Instrument Performance Evaluation Check must be analyzed daily. Acceptance criteria and corrective action are discussed in Section 10.3.1.4 and Appendix 1.

11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.5 and Appendix 1.

**11.3. Method QC**

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid LOD and LOQ must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis. Check Appendix 1 for acceptance criteria.

11.3.5. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

**12.0 CORRECTIVE ACTION**

12.1. Corrective actions associated with this analytical procedure are described in the Summary of In-House Quality Control Procedures in Appendix 1. Document out-of-control event/s and corrective action in the analytical logbook. If the problem persists, consult the supervisor.

12.2. If PEM failed to meet the DDT and Endrin breakdown acceptance criteria, consider the following suggestions to correct the problem:

12.2.1. Deactivate or replace the injection liner.

12.2.2. Check that the injector nut is leak-free.

12.2.3. If problem persists, inform the Supervisor.

12.3. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:

12.3.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.

12.3.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.

12.3.3. If the highest ICAL point appears to be saturated, drop the highest point.

12.3.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

## STANDARD OPERATING PROCEDURE

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*Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.*

- 12.3.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
  - 12.3.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
  - 12.3.5.2. Check the gas flow.
  - 12.3.5.3. Prepare a fresh standard and repeat calibration.
- 12.3.6. If the problem persists, inform the supervisor.
- 12.4. If the ICV is non-compliant, consider the following suggestions to correct the problem:
  - 12.4.1. Re-analyze ICV (to rule out poor injection).
  - 12.4.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error
  - 12.4.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
  - 12.4.4. If the problem persists, inform the supervisor.
- 12.5. If the instrument blank is non-compliant, consider the following suggestions to correct problem:
  - 12.5.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new auto sampler syringe.
  - 12.5.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
  - 12.5.3. Rule out preparation contamination by preparing a new instrument blank.
  - 12.5.4. If the problem persists, inform the supervisor.
- 12.6. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:
  - 12.6.1. Change the liner.
  - 12.6.2. Clean injection port.
  - 12.6.3. Prepare new standard.
  - 12.6.4. Cut or replace column.
  - 12.6.5. Clean the detector.
  - 12.6.6. Rule out leaks by checking all connections.
  - 12.6.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL.
- 12.7. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
  - 12.7.1. Rule out instrument contamination by checking instrument blank.
  - 12.7.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01.
  - 12.7.3. Rule out glassware contamination used for extraction as described in EMAX-QC07.

## STANDARD OPERATING PROCEDURE

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- 12.7.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents.
  - 12.7.5. If the problem persists, inform the supervisor.
  - 12.8. If LCS is non-compliant, perform the following suggestions to correct the problem:
    - 12.8.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level.
    - 12.8.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.
    - 12.8.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.
  - 12.9. Execute a Non-Conformance Report (NCR) when the following circumstances occur:
    - 12.9.1. If corrective action needs the function of other department; e.g., if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.
    - 12.9.2. If corrective action needs the assistance of the project manager; e.g. If the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.
  - 12.10. For other problems encountered, inform the supervisor immediately for further instructions.

**13.0 POLLUTION PREVENTION**

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.
- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

**14.0 WASTE MANAGEMENT**

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

- 15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
  - 15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
  - 15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include

## STANDARD OPERATING PROCEDURE

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samples originating from various matrices, preparation batches, and can exceed 20 samples.

- 15.1.3. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.1.4. Limit of Detection (LOD) – is defined as the smallest amount or concentration of substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1%.
- 15.1.5. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.1.6. Safety Data Sheet (SDS) – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.9. Carry-over – are contaminants retained in the instrument/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).
- 15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical process to monitor contamination.
- 15.1.12. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.13. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.15. Sample Duplicate – is a replicate of a sub-sample taken one sample, prepared and analyzed within the same preparation batch.
- 15.1.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.17. Matrix – A component or form of a sample.

## STANDARD OPERATING PROCEDURE

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15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

15.1.20. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.

15.1.21. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.1.22. Reagent Soil – organic-free Ottawa sand or equivalent.

**15.2. Application of QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing organochlorine pesticides analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

**15.3. Department of Defense (DoD) Projects and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

**16.0 REFERENCES**

- 16.1. "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods", EPA Publication SW-846 Methods 8000B and 8081B, as updated
- 16.2. EMAX Quality Systems Manual, as updated

**17.0 APPENDICES****17.1. Figures**

- |         |          |  |
|---------|----------|--|
| 17.1.1. | Figure 1 | Peak Evaluation Technique                      |
| 17.1.2. | Figure 2 | Typical Chromatogram                           |
| 17.1.3. | Figure 3 | Typical Initial Calibration Summary            |
| 17.1.4. | Figure 4 | Typical Retention Time Window Summary          |
| 17.1.5. | Figure 5 | Typical PEM PEST Breakdown Calculation Summary |
| 17.1.6. | Figure 6 | Typical Sample Result Summary                  |
| 17.1.7. | Figure 7 | Typical LCS Report Summary                     |
| 17.1.8. | Figure 8 | Typical MS/MSD Report Summary                  |
| 17.1.9. | Figure 9 | Typical Case Narrative                         |

**17.2. Tables**

- |         |         |   |
|---------|---------|---|
| 17.2.1. | Table 1 | ICAL Concentration of Individual Analytes   |
| 17.2.2. | Table 2 | ICAL Standard Preparation                   |
| 17.2.3. | Table 3 | Intermediate Primary Standard Preparation   |
| 17.2.4. | Table 4 | Intermediate Secondary Standard Preparation |

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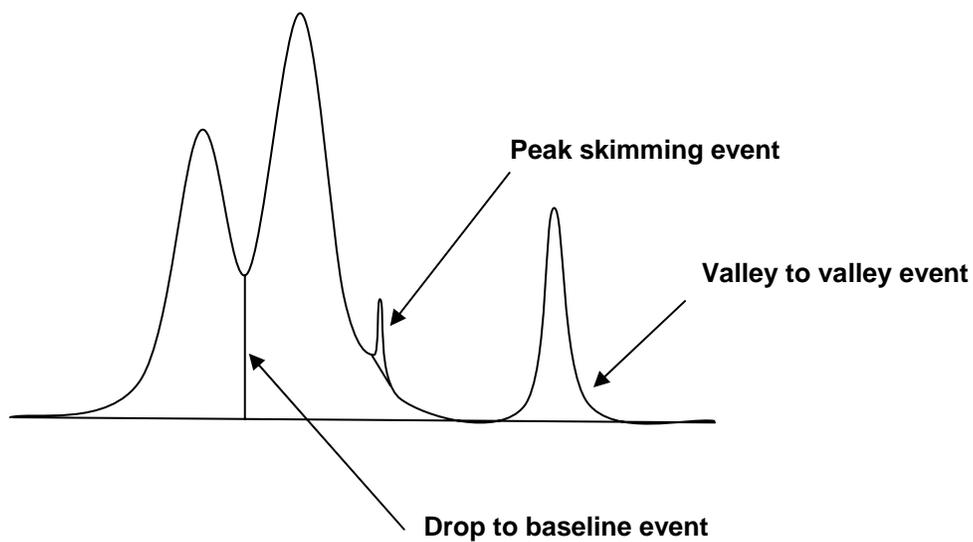
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17.2.5.	Table 5	Check Standard Preparation
17.2.6.	Table 6	Surrogate Standard Preparation
17.2.7.	Table 7	Spike Standard Preparation
17.2.8.	Table 8	Performance Evaluation Mixture Preparation
17.2.9.	Table 9	CLP Retention Time Window for Pesticides
17.2.10.	Table 10	Established Limit of Detection (LOD) & Limit of Quantitation (LOQ)
17.3.	<b>Appendices</b>	
17.3.1.	Appendix 1	Summary of In-House Quality Control Procedures
17.3.2.	Appendix 2	Demonstration of Capability
17.4.	<b>Forms</b>	
17.4.1.	8081FS	Sample Preparation Log
17.4.2.	8081FA	Analytical Run Log
17.4.3.	8081FM	Instrument Maintenance Log

**Figure 1: PEAK EVALUATION TECHNIQUE**

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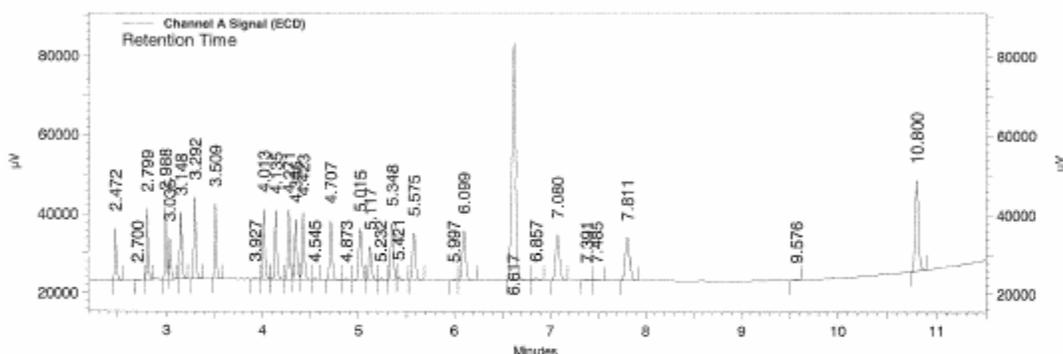


**Figure 2: TYPICAL CHROMATOGRAM**

**Organochlorine Pesticides by GC/ECD**  
**EMAX Laboratories, Inc.**

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Sample ID: ICPE8A2601 (20/200ppb PEST ICV)  
Instrument ID: E8  
Method Name: C:\EZChrom Elite\Methods\CPE8A26.met  
Data: C:\EZChrom Elite\Data\MA26\MA26034.dat  
User: Enrico  
Acquired: 01/27/14 06:35:24  
Printed: 01/27/14 11:45:07



Channel A Signal  
(ECD) Results

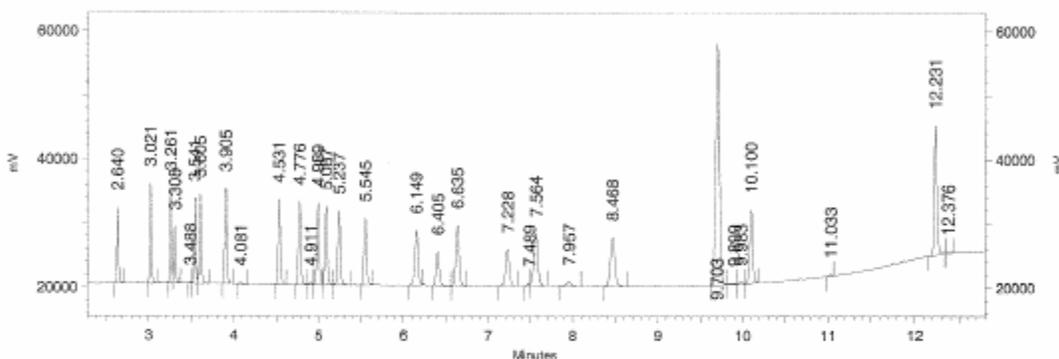
Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration (ppb)	Integration Codes
TCX	2.476	2.472	955775	47593.1	20.082	BB
alpha-BHC	2.804	2.799	1157551	58657.7	19.734	BB
gamma-BHC	2.993	2.988	1194590	60273.4	19.820	BV
beta-BHC	3.040	3.035	755069	37664.5	20.047	VI
delta-BHC	3.153	3.148	1136840	57926.2	19.626	BB
Heptachlor	3.297	3.292	1538033	76393.2	20.133	BB
Aldrin	3.515	3.509	1420481	70593.4	20.122	BB
Heptachlor Epoxide	4.020	4.013	1522180	75468.6	20.170	VV
gamma-Chlordane	4.141	4.135	1539037	76628.8	20.084	VV
alpha-Chlordane	4.277	4.271	1556812	76507.4	20.349	VV
DDE	4.352	4.345	1385456	71405.8	19.403	VV
Endosulfan I	4.429	4.423	1586424	81710.0	19.415	VV
Dieldrin	4.713	4.707	1462708	72989.9	20.040	BV
Endrin	5.021	5.015	1372902	69755.1	19.682	BV
DDD	5.124	5.117	888487	47187.6	18.829	VV
Endosulfan II	5.356	5.348	1607919	77469.6	20.755	BV
DDT	5.581	5.575	1409455	61818.5	22.800	BB
Endrin aldehyde	6.107	6.099	1682493	81300.8	20.695	VB
Methoxychlor	6.625	6.617	8917026	42300.6	210.801	BV
Endosulfan Sulfate	7.087	7.080	1781217	86351.4	20.628	BB
Endrin Ketone	7.820	7.811	1892134	97359.1	19.435	BB
DCB	10.807	10.800	2835411	137403.8	20.636	BB

**Figure 2: TYPICAL CHROMATOGRAM**

**Organochlorine Pesticides by GC/ECD  
EMAX Laboratories, Inc.**

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Sample ID: ICPE8A2601 (20/200ppb PEST ICV)  
Instrument ID: E8  
Method Name: C:\EZChrom Elite\Methods\CPE8A26.met  
Data: C:\EZChrom Elite\Data\MA26\MA26034.dat  
User: Enrico  
Acquired: 01/27/14 06:35:24  
Printed: 01/27/14 11:45:07



**Channel B Signal  
(ECD) Results**

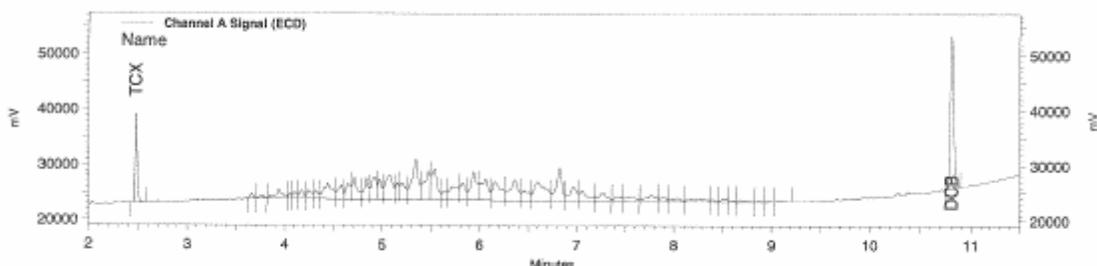
Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration (ppb)	Integration Codes
TCX	2.644	2.640	883518	43071.9	20.513	BB
alpha-BHC	3.027	3.021	1073354	54398.4	19.731	BB
gamma-BHC	3.267	3.261	1035320	52652.3	19.663	BV
beta-BHC	3.313	3.308	674615	33191.9	20.325	VB
delta-BHC	3.547	3.541	977515	51049.0	19.149	VV
Heptachlor	3.612	3.605	1160948	59362.2	19.557	VB
Aldrin	3.911	3.905	1240610	62034.4	19.999	BB
Heptachlor Epoxide	4.537	4.531	1305228	65794.6	19.838	BB
gamma-Chlordane	4.783	4.776	1339776	67877.6	19.738	BV
alpha-Chlordane	4.995	4.989	1366492	68399.9	19.978	VV
Endosulfan I	5.092	5.087	1332936	67777.1	19.666	VV
DDE	5.245	5.237	1314457	67645.1	19.432	VB
Dieldrin	5.552	5.545	1244362	63414.0	19.623	BB
Endrin	6.156	6.149	1182887	59723.1	19.806	BB
DDD	6.412	6.405	734238	38299.0	19.171	BB
Endosulfan II	6.641	6.635	1371009	66625.0	20.578	BB
DDT	7.235	7.228	959460	44911.8	21.363	BB
Endrin Aldehyde	7.571	7.564	1433036	69406.3	20.647	VB
Endosulfan Sulfate	8.476	8.468	1515182	72700.4	20.841	BB
Methoxychlor	9.708	9.703	5835344	26720.6	218.384	BV
Endrin Ketone	10.107	10.100	1577755	79509.5	19.844	BB
DCB	12.236	12.231	2256977	108283.0	20.843	BV

**Figure 2: TYPICAL CHROMATOGRAM**

EPA 8081 by GC/ECD  
 EMAX Analytical Laboratories, Inc.

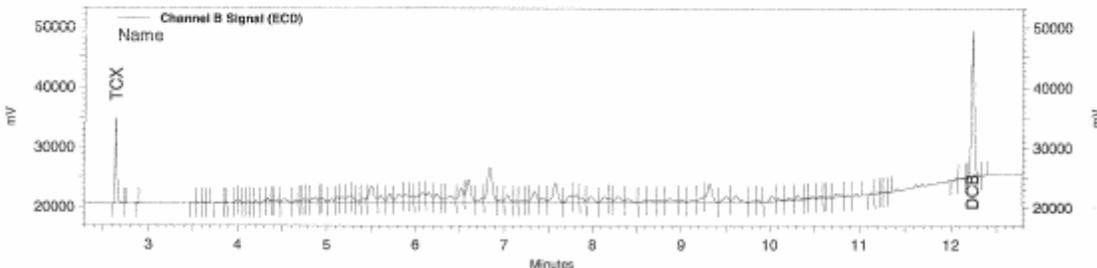
Page 1 of 1

Sample ID: ITOE8A2601 (500/25ppb TOXA ICV)  
 Instrument ID: E8  
 Method Name: C:\EZChrom Elite\Methods\TOE8A26.met  
 Data: C:\EZChrom Elite\Data\MA26\MA26044.dat  
 User: Enrico  
 Acquired: 01/27/14 09:57:27  
 Printed: 01/27/14 14:58:32



Channel A Signal  
 (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.480	2.473	1169230	47804.9	24.46	BB
DCB	10.813	10.804	3396622	139579.7	24.33	IB
Toxaphene			26342693	57718.4	456.40	



Channel B Signal  
 (ECD) Results

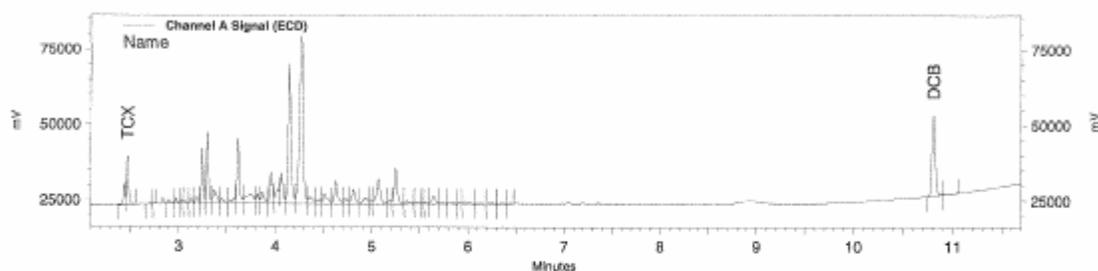
Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.648	2.641	1055995	42950.1	24.59	BV
DCB	12.243	12.235	2709359	112370.7	24.11	VV
Toxaphene			16053757	37656.5	426.32	

**Figure 2: TYPICAL CHROMATOGRAM**

EPA 8081 by GC/ECD  
 EMAX Analytical Laboratories, Inc.

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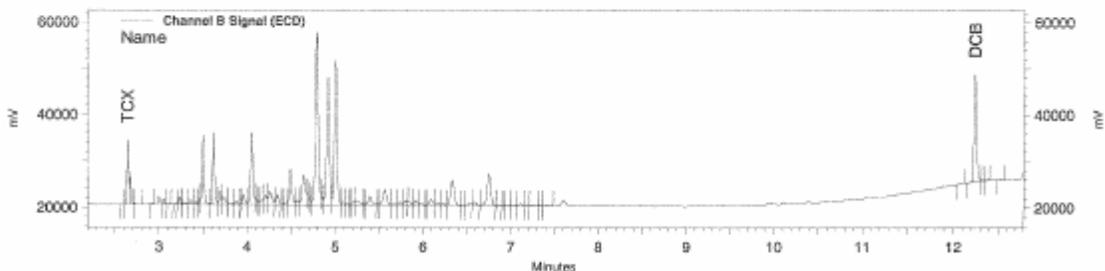
Sample ID: ICRE8A2601 (500/25ppb CHLO ICAL)  
 Instrument ID: E8  
 Method Name: C:\EZChrom Elite\Methods\CRE8A26.met  
 Data: C:\EZChrom Elite\Data\MA26\MA26017.dat  
 User: Enrico  
 Acquired: 01/27/14 00:47:35  
 Printed: 01/27/14 15:39:10



Channel A Signal  
 (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.475	2.475	1186519	56032.1	25.24	VB
DCB	10.805	10.808	3266020	136656.8	23.90	BV

Chlordane 31698308 70804.8 447.69



Channel B Signal  
 (ECD) Results

Name	Expected RT (mins)	Retention Time (mins)	Area	Average RF	ESTD concentration	Integration Codes
TCX	2.643	2.643	955172	47673.5	24.89	SV
DCB	12.233	12.236	2585430	111115.9	23.27	SS

Chlordane 25015410 55675.2 449.31

**Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
METHOD 8081

Lab Name : EMAX Inc  
Instrument ID : E8 (PE CLARUS680 GC)  
GC Column : STX-CLPESTICIDES  
Column size ID : 30MX0.32MMIDXD.32UM  
LFID & Datetime: MA26018A 01/27/14 01:07 MA26026A 01/27/14 03:51  
LFID & Datetime: MA26019A 01/27/14 01:28 MA26027A 01/27/14 04:11  
LFID & Datetime: MA26020A 01/27/14 01:48 MA26028A 01/27/14 04:32  
LFID & Datetime: MA26021A 01/27/14 02:08 MA26029A 01/27/14 04:52  
LFID & Datetime: MA26022A 01/27/14 02:29 MA26030A 01/27/14 05:13  
LFID & Datetime: MA26023A 01/27/14 02:49 MA26031A 01/27/14 05:33  
LFID & Datetime: MA26024A 01/27/14 03:10 MA26032A 01/27/14 05:54  
LFID & Datetime: MA26025A 01/27/14 03:30 MA26033A 01/27/14 06:14  
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS					(AREA)/UNIT				MEAN	%RSD
		1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X			
alpha-BHC	1.25	50461	49985	49796	51003	58714	65922	69878	73502	58657.7	16.8	
Hexachlorobenzene	1.25	151680	148744	145587	138470	133198	123809	116985	114133	134075.9	10.8	
gamma-BHC	1.25	55418	53472	53056	53869	60438	65819	68546	71570	60273.4	12.4	
beta-BHC	1.25	39043	38406	38222	36583	37752	37503	36734	37073	37664.5	2.3	
delta-BHC	1.25	51082	49967	49089	50261	57320	64522	68615	72553	57926.2	16.2	
Heptachlor	1.25	75430	75028	75331	73383	76309	78158	78010	79497	76393.2	2.6	
Aldrin	1.25	67579	66786	65338	65515	70864	74279	76074	78312	70593.4	7.2	
Heptachlor Epoxide	1.25	78332	76078	74193	72576	75070	75350	75340	76810	75468.6	2.3	
gamma-Chlordane	1.25	81370	76728	75232	72774	75604	76192	76714	78415	76628.8	3.3	
alpha-Chlordane	1.25	76556	75072	74055	72745	76650	78084	78783	80113	76507.4	3.2	
DDE	2.50	71592	67348	66695	65767	71126	74599	76133	77986	71405.8	6.4	
Endosulfan I	1.25	84110	81668	80093	78406	81687	82227	82191	83299	81710.0	2.2	
Dieldrin	2.50	69740	68141	67673	67755	74240	77494	78779	80097	72989.9	7.2	
Endrin	2.50	71365	68714	66974	65349	69459	71328	71420	73432	69755.1	3.8	
DDD	2.50	41701	41991	41886	42268	47206	51556	54297	56596	47187.6	13.1	
Endosulfan II	2.50	78419	77252	75515	74694	78285	78634	78291	78667	77469.6	2.0	
DDT	2.50	60030	58843	58134	57951	61977	64277	65890	67446	61818.5	6.0	
Endrin Aldehyde	2.50	90040	86147	83229	80437	80903	78039	76352	75260	81300.8	6.2	
Methoxychlor	12.50	46278	44926	43915	42720	43071	40728	38661	38106	42300.6	6.9	
Endosulfan Sulfate	2.50	91863	88645	86689	85372	86467	84607	83791	83376	86351.4	3.3	
Endrin Ketone	2.50	97937	95853	94346	93656	98673	99419	99172	99816	97359.1	2.5	
Oxychlordane	1.25	80110	76552	74949	70885	69034	65794	63498	63506	70540.9	8.8	
2,4'-DDE	1.25	61390	61447	61948	59722	59374	58300	57066	58216	59682.8	3.0	
trans-Nonachlor	1.25	99849	98909	97932	94988	94271	92067	89873	91036	94865.7	4.0	
2,4'-DDD	1.25	45894	46222	47460	46120	46089	45603	45404	46864	46207.1	1.4	
2,4'-DDT	1.25	63857	63837	63385	61709	61841	60822	59872	61548	62108.8	2.3	
cis-Nonachlor	1.25	103410	104959	101085	96481	95137	93261	91561	93833	97465.8	5.2	
Mirex	1.25	124772	123651	121188	114831	109357	101324	95348	94069	110567.4	11.3	
SURROGATE	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD	
TCX	1.25	50050	49210	47871	46818	47525	46713	46347	46212	47593.1	2.9	
DCB	1.25	150585	148308	144355	140284	139179	130342	123677	122500	137403.8	7.8	

**Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD 8081

Lab Name : EMAX Inc  
 Instrument ID : E8 (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES2  
 Column size ID : 30MX0.32MMIDX0.25UM  
 LFID & Datetime: MA26018B 01/27/14 01:07 MA26026B 01/27/14 03:51  
 LFID & Datetime: MA26019B 01/27/14 01:28 MA26027B 01/27/14 04:11  
 LFID & Datetime: MA26020B 01/27/14 01:48 MA26028B 01/27/14 04:32  
 LFID & Datetime: MA26021B 01/27/14 02:08 MA26029B 01/27/14 04:52  
 LFID & Datetime: MA26022B 01/27/14 02:29 MA26030B 01/27/14 05:13  
 LFID & Datetime: MA26023B 01/27/14 02:49 MA26031B 01/27/14 05:33  
 LFID & Datetime: MA26024B 01/27/14 03:10 MA26032B 01/27/14 05:54  
 LFID & Datetime: MA26025B 01/27/14 03:30 MA26033B 01/27/14 06:14  
 CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS								(AREA)/UNIT	
		1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
alpha-BHC	1.25	50646	48650	45661	45897	51758	60345	64329	67899	54398.4	15.8
Hexachlorobenzene	1.25	142667	129534	130501	124881	119255	112893	107022	104412	121395.5	10.7
gamma-BHC	1.25	45669	45113	44964	46196	52559	59047	62269	65403	52652.3	16.1
beta-BHC	1.25	31707	31626	31893	32056	34007	34380	34498	35369	33191.9	4.6
delta-BHC	1.25	46881	43645	42802	42935	48916	56753	61137	65324	51049.0	17.3
Heptachlor	1.25	64469	60382	56873	54863	57269	59256	60104	61682	59362.2	5.1
Aldrin	1.25	57630	56940	55957	56535	61716	66453	68960	72085	62034.4	10.2
Heptachlor Epoxide	1.25	67663	65619	63343	62127	65151	66233	67036	69183	65794.5	3.5
gamma-Chlordane	1.25	76197	66656	64093	63110	65815	67298	68580	71272	67877.6	6.2
alpha-Chlordane	1.25	70136	67140	65002	63977	67396	69438	70904	73206	68399.9	4.5
DDE	2.50	68214	64858	62697	62248	66686	70036	72249	74172	67645.1	6.4
Endosulfan I	1.25	66117	65987	64767	64418	67911	69585	70883	72549	67777.1	4.4
Dieldrin	2.50	58155	57964	57460	58302	63671	68435	70661	72665	63414.0	10.0
Endrin	2.50	60556	59309	57503	56433	59308	60591	60787	63298	59723.1	3.5
DDD	2.50	34514	33973	34920	34687	37768	40811	43220	46498	38299.0	12.3
Endosulfan II	2.50	65007	64584	63685	63806	67073	68650	69168	71027	66625.0	4.2
DDT	2.50	42030	43174	42810	42513	44950	46480	47650	49689	44911.9	6.2
Endrin Aldehyde	2.50	72006	71214	69592	68371	70244	68647	67376	67801	69406.3	2.4
Methoxychlor	12.50	27839	26742	26456	26189	27140	26720	26095	26583	26720.6	2.1
Endosulfan Sulfate	2.50	73417	74342	72545	71104	72952	72408	71798	73038	72700.4	1.4
Endrin Ketone	2.50	79128	76695	75503	76098	80835	82285	82088	83444	79509.5	3.9
Oxychlorodane	1.25	64121	62089	64713	61264	56762	55132	54054	54658	59099.1	7.5
2,4'-DDE	1.25	70189	62449	62560	58101	56371	54969	53988	55432	59257.5	9.3
trans-Nonachlor	1.25	99838	95641	90381	84884	83673	82103	81026	82847	87549.1	8.0
2,4'-DDD	1.25	37678	36580	38190	39037	38970	39056	38826	40355	38586.6	2.9
2,4'-DDT	1.25	47592	47648	48035	46663	46259	45738	45245	46948	46766.0	2.1
cis-Nonachlor	1.25	81732	82641	83614	82015	81911	81936	81389	84595	82479.3	1.3
Mirex	1.25	103786	105295	105696	98553	93488	86478	81497	80940	94466.8	11.1
SURROGATE	X	1.00X	2.00X	4.00X	8.00X	16.00X	32.00X	48.00X	64.00X	MEAN	%RSD
TCX	1.25	43237	44796	44334	42283	43035	42766	42017	42107	43071.9	2.4
DCB	1.25	119147	116443	112721	109164	108641	103690	99108	97351	108283.0	7.2

**Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD 8081/608

Lab Name : EMAX Inc  
 Instrument ID : EB (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES  
 Column size ID : 30MX0.32MMIDX0.32UM  
 LFD & Datetime: MA26036A 01/27/14 07:16  
 LFD & Datetime: MA26037A 01/27/14 07:36  
 LFD & Datetime: MA26038A 01/27/14 07:56  
 LFD & Datetime: MA26039A 01/27/14 08:16  
 LFD & Datetime: MA26040A 01/27/14 08:36  
 LFD & Datetime: MA26041A 01/27/14 08:57  
 LFD & Datetime: MA26042A 01/27/14 09:17  
 LFD & Datetime: MA26043A 01/27/14 09:37  
 CONC UNIT: ppb

COMPOUND	CONC x	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
		1.00x	2.00x	5.00x	10.00x	15.00x	20.00x	30.00x	40.00x		
Toxaphene	50.00	58631	53413	56818	57730	58674	58520	58234	59727	57718.4	3.3

INITIAL CALIBRATION  
 METHOD 8081/608

Lab Name : EMAX Inc  
 Instrument ID : EB (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES2  
 Column size ID : 30MX0.32MMIDX0.25UM  
 LFD & Datetime: MA26036B 01/27/14 07:16  
 LFD & Datetime: MA26037B 01/27/14 07:36  
 LFD & Datetime: MA26038B 01/27/14 07:56  
 LFD & Datetime: MA26039B 01/27/14 08:16  
 LFD & Datetime: MA26040B 01/27/14 08:36  
 LFD & Datetime: MA26041B 01/27/14 08:57  
 LFD & Datetime: MA26042B 01/27/14 09:17  
 LFD & Datetime: MA26043B 01/27/14 09:37  
 CONC UNIT: ppb

COMPOUND	CONC x	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
		1.00x	2.00x	5.00x	10.00x	15.00x	20.00x	30.00x	40.00x		
Toxaphene	50.00	38754	36281	36249	36766	37480	37838	38209	39675	37656.5	3.2

**Figure 3: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8081

Lab Name : EMAX Inc  
 Instrument ID : E8 (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES  
 Column size ID : 30MX0.32MMIDX0.32UM  
 LFD & Datetime: MA26009A 01/26/14 22:07  
 LFD & Datetime: MA26010A 01/26/14 22:27  
 LFD & Datetime: MA26011A 01/26/14 22:47  
 LFD & Datetime: MA26012A 01/26/14 23:07  
 LFD & Datetime: MA26013A 01/26/14 23:27  
 LFD & Datetime: MA26014A 01/26/14 23:47  
 LFD & Datetime: MA26015A 01/27/14 00:07  
 LFD & Datetime: MA26016A 01/27/14 00:27  
 CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
		1.00X	2.00X	4.00X	10.00X	20.00X	30.00X	40.00X	60.00X		
CHLORDANE	25.00	89545	68121	80192	70426	67071	64747	63980	62357	70804.9	13.2

Lab Name : EMAX Inc  
 Instrument ID : E8 (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES2  
 Column size ID : 30MX0.32MMIDX0.25UM  
 LFD & Datetime: MA26009B 01/26/14 22:07  
 LFD & Datetime: MA26010B 01/26/14 22:27  
 LFD & Datetime: MA26011B 01/26/14 22:47  
 LFD & Datetime: MA26012B 01/26/14 23:07  
 LFD & Datetime: MA26013B 01/26/14 23:27  
 LFD & Datetime: MA26014B 01/26/14 23:47  
 LFD & Datetime: MA26015B 01/27/14 00:07  
 LFD & Datetime: MA26016B 01/27/14 00:27  
 CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
		1.00X	2.00X	4.00X	10.00X	20.00X	30.00X	40.00X	60.00X		
CHLORDANE	25.00	74070	56360	52924	51998	52846	52524	52477	52202	55675.2	13.6

**Figure 4: TYPICAL ICAL RETENTION TIME WINDOW SUMMARY**

INITIAL CALIBRATION  
 METHOD 8081

Lab Name : EMAX Inc  
 Instrument ID : E8 (PE CLARUS680 GC)  
 GC Column : STX-CLPESTICIDES  
 Column size ID : 30MX0.32MMIDX0.32UM  
 LFID & Datetime: MA26018A 01/27/14 01:07 MA26026A 01/27/14 03:51  
 LFID & Datetime: MA26019A 01/27/14 01:28 MA26027A 01/27/14 04:11  
 LFID & Datetime: MA26020A 01/27/14 01:48 MA26028A 01/27/14 04:32  
 LFID & Datetime: MA26021A 01/27/14 02:08 MA26029A 01/27/14 04:52  
 LFID & Datetime: MA26022A 01/27/14 02:29 MA26030A 01/27/14 05:13  
 LFID & Datetime: MA26023A 01/27/14 02:49 MA26031A 01/27/14 05:33  
 LFID & Datetime: MA26024A 01/27/14 03:10 MA26032A 01/27/14 05:54  
 LFID & Datetime: MA26025A 01/27/14 03:30 MA26033A 01/27/14 06:14

COMPOUND	RT OF STANDARDS (MIN)								MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X		FROM	TO	
alpha-BHC	2.800	2.801	2.800	2.805	2.804	2.804	2.803	2.801	2.802	2.782	2.822	0.020
Hexachlorobenzene	2.704	2.707	2.708	2.699	2.701	2.705	2.705	2.701	2.704	2.684	2.724	0.020
gamma-BHC	2.988	2.991	2.988	2.995	2.993	2.993	2.992	2.989	2.991	2.969	3.013	0.022
beta-BHC	3.036	3.039	3.036	3.043	3.040	3.041	3.039	3.037	3.039	3.017	3.061	0.022
delta-BHC	3.149	3.151	3.148	3.155	3.153	3.155	3.152	3.151	3.152	3.131	3.173	0.021
Heptachlor	3.293	3.295	3.293	3.299	3.297	3.297	3.296	3.293	3.295	3.271	3.319	0.024
Aldrin	3.511	3.512	3.511	3.516	3.515	3.515	3.513	3.511	3.513	3.487	3.539	0.026
Heptachlor Epoxide	4.016	4.019	4.016	4.023	4.020	4.021	4.019	4.016	4.019	3.989	4.049	0.030
gamma-Chlordane	4.137	4.139	4.136	4.143	4.141	4.141	4.140	4.137	4.139	4.107	4.171	0.032
alpha-Chlordane	4.273	4.275	4.272	4.279	4.277	4.277	4.276	4.273	4.275	4.243	4.307	0.032
DDE	4.348	4.349	4.348	4.353	4.352	4.353	4.351	4.348	4.350	4.318	4.382	0.032
Endosulfan I	4.425	4.427	4.425	4.431	4.429	4.429	4.428	4.425	4.427	4.392	4.462	0.035
Dieldrin	4.711	4.712	4.709	4.716	4.713	4.715	4.713	4.709	4.712	4.680	4.744	0.032
Endrin	5.021	5.020	5.019	5.025	5.021	5.024	5.021	5.019	5.021	4.991	5.051	0.030
DDD	5.121	5.121	5.120	5.127	5.124	5.127	5.124	5.121	5.123	5.093	5.153	0.030
Endosulfan II	5.353	5.353	5.352	5.359	5.356	5.357	5.355	5.352	5.355	5.326	5.384	0.029
DDT	5.580	5.580	5.579	5.584	5.581	5.583	5.581	5.579	5.581	5.551	5.611	0.030
Endrin Aldehyde	6.104	6.104	6.103	6.108	6.107	6.107	6.104	6.101	6.105	6.073	6.137	0.032
Methoxychlor	6.624	6.623	6.624	6.629	6.625	6.628	6.624	6.620	6.625	6.587	6.663	0.038
Endosulfan Sulfate	7.085	7.085	7.085	7.091	7.087	7.089	7.085	7.083	7.086	7.050	7.122	0.036
Endrin Ketone	7.817	7.819	7.817	7.821	7.820	7.820	7.817	7.815	7.818	7.782	7.854	0.036
Oxychlordane	3.925	3.927	3.927	3.919	3.921	3.925	3.925	3.921	3.924	3.905	3.943	0.019
2,4'-DDE	3.999	4.000	4.000	3.992	3.995	4.000	4.000	3.995	3.998	3.976	4.020	0.022
trans-Nonachlor	4.257	4.259	4.259	4.251	4.253	4.259	4.259	4.253	4.256	4.233	4.279	0.023
2,4'-DDD	4.551	4.552	4.552	4.544	4.547	4.551	4.552	4.545	4.549	4.524	4.574	0.025
2,4'-DDT	4.880	4.880	4.880	4.872	4.875	4.879	4.880	4.875	4.878	4.855	4.901	0.023
cis-Nonachlor	5.072	5.072	5.072	5.064	5.067	5.071	5.072	5.067	5.070	5.048	5.092	0.022
Mirex	6.788	6.788	6.788	6.781	6.783	6.785	6.788	6.781	6.785	6.757	6.813	0.028
SURROGATE	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
TCX	2.472	2.475	2.472	2.477	2.476	2.476	2.475	2.473	2.474	2.454	2.494	0.020
DCB	10.804	10.804	10.803	10.807	10.807	10.805	10.801	10.801	10.804	10.754	10.854	0.050

**Figure 4: TYPICAL ICAL RETENTION TIME WINDOW SUMMARY**

INITIAL CALIBRATION  
METHOD 8081

Lab Name : EMAX Inc  
Instrument ID : E8 (PE CLARUS680 GC)  
GC Column : STX-CLPESTICIDES2  
Column size ID : 30MX0.32MMIDX0.25UM  
LFID & Datetime: MA26018B 01/27/14 01:07 MA26026B 01/27/14 03:51  
LFID & Datetime: MA26019B 01/27/14 01:28 MA26027B 01/27/14 04:11  
LFID & Datetime: MA26020B 01/27/14 01:48 MA26028B 01/27/14 04:32  
LFID & Datetime: MA26021B 01/27/14 02:08 MA26029B 01/27/14 04:52  
LFID & Datetime: MA26022B 01/27/14 02:29 MA26030B 01/27/14 05:13  
LFID & Datetime: MA26023B 01/27/14 02:49 MA26031B 01/27/14 05:33  
LFID & Datetime: MA26024B 01/27/14 03:10 MA26032B 01/27/14 05:54  
LFID & Datetime: MA26025B 01/27/14 03:30 MA26033B 01/27/14 06:14

COMPOUND	RT OF STANDARDS (MIN)								MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X		FROM	TO	
alpha-BHC	3.021	3.024	3.023	3.029	3.027	3.027	3.025	3.024	3.025	3.007	3.043	0.018
Hexachlorobenzene	2.941	2.944	2.945	2.936	2.939	2.944	2.943	2.939	2.941	2.923	2.959	0.018
gamma-BHC	3.261	3.264	3.261	3.268	3.267	3.267	3.264	3.263	3.264	3.247	3.281	0.017
beta-BHC	3.308	3.311	3.309	3.316	3.313	3.313	3.312	3.309	3.311	3.294	3.328	0.017
delta-BHC	3.541	3.545	3.543	3.549	3.547	3.547	3.548	3.545	3.544	3.545	3.528	0.017
Heptachlor	3.607	3.611	3.608	3.615	3.612	3.612	3.609	3.608	3.610	3.592	3.628	0.018
Aldrin	3.907	3.909	3.908	3.913	3.911	3.912	3.909	3.908	3.910	3.891	3.929	0.019
Heptachlor Epoxide	4.533	4.536	4.533	4.540	4.537	4.539	4.536	4.533	4.536	4.513	4.559	0.023
gamma-Chlordane	4.780	4.783	4.780	4.785	4.783	4.785	4.781	4.779	4.782	4.761	4.803	0.021
alpha-Chlordane	4.992	4.993	4.992	4.999	4.995	4.997	4.995	4.991	4.994	4.976	5.012	0.018
DDE	5.241	5.244	5.241	5.248	5.245	5.247	5.244	5.241	5.244	5.228	5.260	0.016
Endosulfan I	5.089	5.092	5.089	5.096	5.092	5.095	5.093	5.089	5.092	5.073	5.111	0.019
Dieldrin	5.548	5.551	5.549	5.556	5.552	5.555	5.551	5.548	5.551	5.534	5.568	0.017
Endrin	6.155	6.156	6.155	6.161	6.156	6.159	6.156	6.153	6.156	6.136	6.176	0.020
DDD	6.409	6.412	6.411	6.416	6.412	6.415	6.412	6.408	6.412	6.393	6.431	0.019
Endosulfan II	6.640	6.641	6.639	6.645	6.641	6.644	6.641	6.636	6.641	6.619	6.663	0.022
DDT	7.232	7.235	7.233	7.240	7.235	7.237	7.235	7.231	7.235	7.211	7.259	0.024
Endrin Aldehyde	7.568	7.571	7.569	7.575	7.571	7.573	7.569	7.567	7.570	7.548	7.592	0.022
Methoxychlor	9.704	9.707	9.705	9.711	9.708	9.709	9.705	9.703	9.707	9.679	9.734	0.028
Endosulfan Sulfate	8.473	8.476	8.475	8.479	8.476	8.479	8.475	8.471	8.476	8.451	8.500	0.024
Endrin Ketone	10.103	10.105	10.104	10.108	10.107	10.108	10.104	10.101	10.105	10.075	10.135	0.030
Oxychlordane	4.419	4.420	4.421	4.413	4.415	4.420	4.420	4.415	4.418	4.398	4.438	0.020
2,4'-DDE	4.769	4.771	4.772	4.764	4.767	4.771	4.771	4.767	4.769	4.748	4.790	0.021
trans-Nonachlor	4.908	4.908	4.909	4.901	4.904	4.908	4.909	4.904	4.906	4.888	4.924	0.018
2,4'-DDD	5.611	5.612	5.612	5.604	5.607	5.611	5.612	5.607	5.609	5.591	5.627	0.018
2,4'-DDT	6.243	6.243	6.244	6.236	6.239	6.244	6.244	6.240	6.242	6.223	6.261	0.019
cis-Nonachlor	6.328	6.328	6.329	6.321	6.324	6.329	6.329	6.325	6.327	6.309	6.345	0.018
Mirex	9.928	9.929	9.927	9.921	9.923	9.928	9.927	9.924	9.926	9.902	9.950	0.024
SURROGATE	1.0X	2.0X	4.0X	8.0X	16.0X	32.0X	48.0X	64.0X	RT	FROM	TO	WIDTH
TCX	2.640	2.643	2.640	2.647	2.644	2.644	2.643	2.641	2.643	2.620	2.666	0.023
DCB	12.231	12.233	12.232	12.236	12.236	12.235	12.231	12.229	12.233	12.198	12.268	0.035

**Figure 5: TYPICAL PEM PEST BREAKDOWN CALCULATION SUMMARY**

PEM PEST BREAKDOWN CALCULATION  
 METHOD 8081

Lab Name : EMAX  
 Instrument ID : GCT016 HP-5890  
 GC Column : RTX-CLPEST RTX-CLPESTII  
 Column size ID : .32MMX30M .32MMX30M  
 PEM LFID & Datetime : WF17026A WF17026B 06/18/10 00:50

Base on AREA

LFID	AREA			TOTAL	% Breakdown			QL	QCLIMIT
	DDD	DDE	DDT		DDD	DDE	TOTAL		
WF17026A	0.0	11917.0	826713.0	838630.0	0.00	1.42	1.42		15
WF17026B	21368.0	17092.0	933363.0	971823.0	2.20	1.76	3.96		15
LFID	ENDRIN	ENDRIN ALDEHYDE	ENDRIN KETONE	TOTAL	ENDRIN ALDEHYDE	ENDRIN KETONE	TOTAL	QL	QCLIMIT
WF17026A	893765.0	48233.0	35430.0	977428.0	4.93	3.62	8.56		15
WF17026B	945951.0	60214.0	48710.0	1054875.0	5.71	4.62	10.33		15

**Figure 6: TYPICAL SAMPLE RESULT SUMMARY**

METHOD SW3520C/8081A  
 PESTICIDES

Client : XYZ, INC.	Date Collected: 01/15/14
Project : CLEAN PROJECT	Date Received: 01/16/14
Batch No. : 14A051	Date Extracted: 01/21/14 14:00
Sample ID: EMW02012014	Date Analyzed: 01/28/14 18:09
Lab Samp ID: A051-03	Dilution Factor: 1
Lab File ID: MA28020A	Matrix : WATER
Ext Btch ID: CPA024W	% Moisture : NA
Calib. Ref.: MA28011A	Instrument ID : GCE8

PARAMETERS	RESULTS (ug/L)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)
ALPHA-BHC	(ND) ND	0.10	0.0050	0.010
GAMMA-BHC	(ND) ND	0.10	0.0050	0.010
BETA-BHC	(ND) ND	0.10	0.0070	0.010
HEPTACHLOR	(ND) ND	0.10	0.0050	0.010
DELTA-BHC	(ND) ND	0.10	0.0070	0.010
ALDRIN	(ND) ND	0.10	0.0050	0.010
HEPTACHLOR EPOXIDE	(ND) ND	0.10	0.0050	0.010
GAMMA-CHLORDANE	(ND) ND	0.10	0.0050	0.010
ALPHA-CHLORDANE	(ND) ND	0.10	0.0050	0.010
ENDOSULFAN I	(ND) ND	0.10	0.0080	0.010
4,4'-DDE	(ND) ND	0.10	0.0050	0.010
DIELDRIN	(ND) ND	0.10	0.0050	0.010
ENDRIN	(ND) ND	0.10	0.0080	0.010
4,4'-DDD	(ND) ND	0.10	0.0050	0.010
ENDOSULFAN II	(ND) ND	0.10	0.0050	0.010
4,4'-DDT	(ND) ND	0.10	0.0050	0.010
ENDRIN ALDEHYDE	(ND) ND	0.10	0.0050	0.010
ENDOSULFAN SULFATE	(ND) ND	0.10	0.0050	0.010
ENDRIN KETONE	(ND) ND	0.10	0.0050	0.010
METHOXYCHLOR	(ND) ND	1.0	0.050	0.10
TOXAPHENE	(ND) ND	2.0	0.25	0.50
CHLORDANE	(ND) ND	1.0	0.12	0.25

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
TETRACHLORO-M-XYLENE	(0.3346)   0.3344	0.4000	(83.7)   83.6	25-140
DECACHLOROBIPHENYL	(0.4240)   0.4139	0.4000	(106)   103	30-135

RL : Reporting limit  
 Left of | is related to first column ; Right of | related to second column  
 Final result indicated by ( )

Figure 7:

TYPICAL LCS/LCSD REPORT SUMMARY

EMAX QUALITY CONTROL DATA  
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
PROJECT: CLEAN PROJECT  
BATCH NO.: 14A051  
METHOD: SW3520C/8081A

MATRIX: WATER % MOISTURE: NA  
DILUTION FACTOR: 1 1 1  
SAMPLE ID: MBLK1W  
LAB SAMP ID: CPA024WB CPA024WL CPA024WC  
LAB FILE ID: MA28015A MA28016A MA28017A  
DATE EXTRACTED: 01/21/1414:00 01/21/1414:00 01/21/1414:00 DATE COLLECTED: NA  
DATE ANALYZED: 01/28/1416:27 01/28/1416:47 01/28/1417:08 DATE RECEIVED: 01/21/14  
PREP. BATCH: CPA024W CPA024W CPA024W  
CALIB. REF: MA28011A MA28011A MA28011A

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
alpha-BHC	(ND) ND	0.200	(0.203) 0.189	(101) 94	0.200	(0.204) 0.190	(102) 95	(0) 1	60-130	30
gamma-BHC	(ND) ND	0.200	(0.202) 0.198	(101) 99	0.200	(0.202) 0.199	(101) 100	(0) 1	25-135	30
beta-BHC	(ND) ND	0.200	0.206 (0.213)	103 (106)	0.200	0.204 (0.214)	102 (107)	1 (0)	65-125	30
Heptachlor	(ND) ND	0.200	0.204 (0.208)	102 (104)	0.200	0.203 (0.206)	101 (103)	0 (1)	40-130	30
delta-BHC	(ND) ND	0.200	(0.212) 0.204	(106) 102	0.200	(0.210) 0.203	(105) 101	(1) 0	45-135	30
Aldrin	(ND) ND	0.200	(0.210) 0.205	(105) 102	0.200	(0.208) 0.204	(104) 102	(1) 0	25-140	30
Heptachlor Epoxide	(ND) ND	0.200	(0.221) 0.216	(110) 108	0.200	0.212 (0.215)	106 (108)	4 (0)	60-130	30
gamma-Chlordane	(ND) ND	0.200	0.215 (0.218)	108 (109)	0.200	0.213 (0.218)	106 (109)	1 (0)	60-125	30
alpha-Chlordane	(ND) ND	0.200	(0.219) 0.212	(110) 106	0.200	(0.217) 0.210	(108) 105	(1) 1	65-125	30
Endosulfan I	(ND) ND	0.200	(0.208) 0.204	(104) 102	0.200	(0.208) 0.202	(104) 101	(0) 1	50-110	30
4,4'-DDE	(ND) ND	0.200	(0.224) 0.212	(112) 106	0.200	(0.221) 0.207	(110) 104	(1) 2	35-140	30
Dieldrin	(ND) ND	0.200	(0.213) 0.212	(106) 106	0.200	(0.212) 0.209	(106) 104	(0) 1	60-130	30
Endrin	(ND) ND	0.200	(0.228) 0.224	(114) 112	0.200	(0.229) 0.221	(114) 110	(0) 1	55-135	30
4,4'-DDD	(ND) ND	0.200	0.217 (0.227)	108 (114)	0.200	0.213 (0.223)	106 (112)	2 (2)	25-150	30
Endosulfan II	(ND) ND	0.200	(0.215) 0.209	(108) 104	0.200	(0.213) 0.207	(106) 104	(1) 1	30-130	30
4,4'-DDT	(ND) ND	0.200	(0.255) 0.244	(127) 122	0.200	(0.253) 0.242	(126) 121	(1) 1	45-140	30
Endrin aldehyde	(ND) ND	0.200	(0.213) 0.202	(106) 101	0.200	(0.211) 0.208	(105) 104	(1) 3	55-135	30
Endosulfan Sulfate	(ND) ND	0.200	(0.226) 0.219	(113) 110	0.200	(0.225) 0.217	(112) 108	(0) 1	55-135	30
Endrin ketone	(ND) ND	0.200	0.208 (0.211)	104 (105)	0.200	0.207 (0.209)	104 (104)	0 (1)	75-125	30
Methoxychlor	(ND) ND	2.00	2.30 (2.88)	115 (144)	2.00	2.28 (2.68)	114 (134)	1 (7)	55-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT (%)
Tetrachloro-m-xylene	0.4000	(0.3778) 0.3682	(94.4) 92.0	0.4000	(0.3608) 0.3530	(90.2) 88.3	25-140
Decachlorobiphenyl	0.4000	(0.4321) 0.4309	(108) 108	0.4000	(0.4262) 0.4226	(107) 106	30-135

Figure 8:

TYPICAL MS/MSD REPORT SUMMARY

EMAX QUALITY CONTROL DATA  
MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
PROJECT: CLEAN PROJECT  
BATCH NO.: 14A051  
METHOD: SW3520C/8081A

MATRIX: WATER % MOISTURE: NA  
DILUTION FACTOR: 1 1.1 1.02  
SAMPLE ID: EMW02012014  
LAB SAMP ID: A051-03 A051-03M A051-03S  
LAB FILE ID: MA28020A MA28021A MA28022A  
DATE EXTRACTED: 01/21/1414:00 01/21/1414:00 01/21/1414:00 DATE COLLECTED: 01/15/14  
DATE ANALYZED: 01/28/1418:09 01/28/1418:29 01/28/1418:50 DATE RECEIVED: 01/16/14  
PREP. BATCH: CPA024W CPA024W CPA024W  
CALIB. REF: MA28011A MA28011A MA28011A

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)		SPIKE AMT (ug/L)	MS RSLT (ug/L)		MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)		MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )			
alpha-BHC	(ND)	ND	0.220	(0.232)	0.217	(105)	99	0.204	(0.215)	0.201	(105)	99	(8)	8	60-130	30
gamma-BHC	(ND)	ND	0.220	0.227	(0.230)	103	(105)	0.204	(0.211)	0.211	(103)	103	(7)	9	25-135	30
beta-BHC	(ND)	ND	0.220	0.228	(0.253)	104	(115)	0.204	0.210	(0.231)	103	(113)	8	(9)	65-125	30
Heptachlor	(ND)	ND	0.220	0.230	(0.236)	105	(107)	0.204	0.211	(0.217)	103	(106)	9	(8)	40-130	30
delta-BHC	(ND)	ND	0.220	(0.235)	0.234	(107)	106	0.204	(0.217)	0.215	(106)	105	(8)	8	45-135	30
Aldrin	(ND)	ND	0.220	(0.236)	0.236	(107)	107	0.204	(0.218)	0.218	(107)	107	(8)	8	25-140	30
Heptachlor Epoxide	(ND)	ND	0.220	0.241	(0.243)	110	(110)	0.204	(0.233)	0.223	(114)	109	(3)	9	60-130	30
gamma-Chlordane	(ND)	ND	0.220	(0.243)	0.243	(110)	110	0.204	(0.225)	0.225	(110)	110	(8)	8	60-125	30
alpha-Chlordane	(ND)	ND	0.220	(0.249)	0.237	(113)	108	0.204	(0.229)	0.221	(112)	108	(8)	7	65-125	30
Endosulfan I	(ND)	ND	0.220	(0.235)	0.227	(107)	103	0.204	(0.217)	0.211	(106)	103	(8)	7	50-110	30
4,4'-DDE	(ND)	ND	0.220	(0.252)	0.234	(115)	106	0.204	(0.232)	0.219	(114)	107	(8)	7	35-140	30
Dieldrin	(ND)	ND	0.220	(0.245)	0.239	(111)	109	0.204	(0.227)	0.221	(111)	108	(8)	8	60-130	30
Endrin	(ND)	ND	0.220	(0.261)	0.253	(119)	115	0.204	(0.240)	0.235	(118)	115	(8)	7	55-135	30
4,4'-DDD	(ND)	ND	0.220	0.246	(0.256)	112	(116)	0.204	0.227	(0.238)	111	(117)	8	(7)	25-150	30
Endosulfan II	(ND)	ND	0.220	(0.242)	0.235	(110)	107	0.204	(0.225)	0.218	(110)	107	(7)	8	30-130	30
4,4'-DDT	(ND)	ND	0.220	(0.292)	0.278	(133)	126	0.204	(0.271)	0.257	(133)	126	(7)	8	45-140	30
Endrin aldehyde	(ND)	ND	0.220	(0.240)	0.236	(109)	107	0.204	(0.222)	0.214	(109)	105	(8)	10	55-135	30
Endosulfan Sulfate	(ND)	ND	0.220	(0.257)	0.249	(117)	113	0.204	(0.238)	0.230	(117)	113	(8)	8	55-135	30
Endrin Ketone	(ND)	ND	0.220	0.235	(0.240)	107	(109)	0.204	0.220	(0.223)	108	(109)	7	(7)	75-125	30
Methoxychlor	(ND)	ND	2.20	2.62	(3.05)	119	(139)	2.04	2.43	(2.82)	119	(138)	8	(8)	55-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	MS RSLT (ug/L)	MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)	MSD % REC	QC LIMIT ( % )
Tetrachloro-m-xylene	0.4400	(0.4345)   0.4180	(98.7)   95.0	0.4080	(0.3784)   0.3710	(92.7)   90.9	25-140
Decachlorobiphenyl	0.4400	(0.4836)   0.4728	(110)   107	0.4080	(0.4427)   0.4327	(109)   106	30-135

\* : Out side of QC Limit.

Figure 9:

TYPICAL CASE NARRATIVE

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN PROJECT  
SDG : 14A051

METHOD SW3520C/8081A  
PESTICIDES

A total of three (3) water samples were received on 01/16/14 for Pesticides Organochlorine analysis, Method SW3520C/8081A in accordance with Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2 and Project QAPP 5/2012.

Holding Time

Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration

Instrument performance was checked prior to calibration. DDT and Endrin breakdown were within specification. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for CPA024WL/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the samples in this SDG. Percent recoveries for A051-03M/S were within project QC limits.

Surrogate

Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Positive sample results were confirmed by a second column. Relative percentage difference (RPD) between the two results was evaluated. If RPD is less than 40% and peaks are well defined the higher result is reported. Where RPD is greater than 40% the chromatogram is checked for anomalies and results are selected based on processed knowledge. If there is no evidence of any chromatographic ambiguity, the higher result is reported.

**Table 1: ICAL CONCENTRATION OF INDIVIDUAL ANALYTES**

PARAMETERS	ICAL STANDARD CONCENTRATION (µg/L)							
	1	2	3	4	5	6	7	8
<b>PESTICIDES</b>								
Aldrin	1.25	2.5	5	10	20	40	60	80
alpha-BHC	1.25	2.5	5	10	20	40	60	80
beta-BHC	1.25	2.5	5	10	20	40	60	80
delta-BHC	1.25	2.5	5	10	20	40	60	80
gamma-BHC	1.25	2.5	5	10	20	40	60	80
alpha-Chlordane	1.25	2.5	5	10	20	40	60	80
gamma-Chlordane	1.25	2.5	5	10	20	40	60	80
Heptachlor	1.25	2.5	5	10	20	40	60	80
Heptachlor Epoxide	1.25	2.5	5	10	20	40	60	80
Endosulfan I	1.25	2.5	5	10	20	40	60	80
4,4' – DDD	2.5	5	10	20	40	80	120	160
4,4' – DDE	2.5	5	10	20	40	80	120	160
4,4' – DDT	2.5	5	10	20	40	80	120	160
Dieldrin	2.5	5	10	20	40	80	120	160
Endrin	2.5	5	10	20	40	80	120	160
Endosulfan II	2.5	5	10	20	40	80	120	160
Endosulfan Sulfate	2.5	5	10	20	40	80	120	160
Endrin Aldehyde	2.5	5	10	20	40	80	120	160
Endrin Ketone	2.5	5	10	20	40	80	120	160
Methoxychlor	12.5	25	50	100	200	400	600	800
2,4' – DDD	1.25	2.5	5	10	20	40	60	80
2,4' – DDE	1.25	2.5	5	10	20	40	60	80
2,4' – DDT	1.25	2.5	5	10	20	40	60	80
Oxychlordane	1.25	2.5	5	10	20	40	60	80
cis-Nonachlor	1.25	2.5	5	10	20	40	60	80
Trans-Nonachlor	1.25	2.5	5	10	20	40	60	80
Mirex	1.25	2.5	5	10	20	40	60	80
Tetrachloro-m-xylene (surrogate)	1.25	2.5	5	10	20	40	60	80
Decachlorobiphenyl (Surrogate)	1.25	2.5	5	10	20	40	60	80
TOXAPHENE	50	100	250	500	750	1000	1500	2000
TECHNICAL CHLORDANE	25	100	250	500	750	1000	1500	2000

**Table 2: ICAL STANDARD PREPARATION**

Standard #	Compound Name	Intermediate Solution Conc. (µg/L)	Preparation			Final Conc. (µg/L)
			Aliquot (µL)	Solvent	Final Volume (µL)	
<b>PESTICIDES</b>						
1	Pest ICAL Mix1	80 / 160 / 800	12.5	Hexane	800	1.25 / 2.5 / 12.5
2	Pest ICAL Mix1	80 / 160 / 800	25	Hexane	800	2.5 / 5 / 25
3	Pest ICAL Mix1	80 / 160 / 800	50	Hexane	800	5 / 10 / 50
4	Pest ICAL Mix1	80 / 160 / 800	100	Hexane	800	10 / 20 / 100
5	Pest ICAL Mix1	80 / 160 / 800	200	Hexane	800	20 / 40 / 200
6	Pest ICAL Mix1	80 / 160 / 800	400	Hexane	800	40 / 80 / 400
7	Pest ICAL Mix1	80 / 160 / 800	600	Hexane	800	60 / 120 / 600
8	Pest ICAL Mix1	80 / 160 / 800	800	-	800	80 / 160 / 800
<b>TOXAPHENE</b>						
1	Toxaphene ICAL	2000	20	Hexane	800	50
2	Toxaphene ICAL	2000	40	Hexane	800	100
3	Toxaphene ICAL	2000	100	Hexane	800	250
4	Toxaphene ICAL	2000	200	Hexane	800	500
5	Toxaphene ICAL	2000	300	Hexane	800	750
6	Toxaphene ICAL	2000	400	Hexane	800	1000
7	Toxaphene ICAL	2000	600	Hexane	800	1500
8	Toxaphene ICAL	2000	800	-	800	2000
<b>TECHNICAL CHLORDANE</b>						
1	Tech. Chlordane ICAL	2000	10	Hexane	800	25
2	Tech. Chlordane ICAL	2000	40	Hexane	800	100
3	Tech. Chlordane ICAL	2000	100	Hexane	800	250
4	Tech. Chlordane ICAL	2000	200	Hexane	800	500
5	Tech. Chlordane ICAL	2000	300	Hexane	800	750
6	Tech. Chlordane ICAL	2000	400	Hexane	800	1000
7	Tech. Chlordane ICAL	2000	600	Hexane	800	1500
8	Tech. Chlordane ICAL	2000	800	-	800	2000
<b>PESTICIDES MIX 3</b>						
1	Pest ICAL Mix 3	80	12.5	Hexane	800	1.25
2	Pest ICAL Mix 3	80	25	Hexane	800	2.5
3	Pest ICAL Mix 3	80	50	Hexane	800	5
4	Pest ICAL Mix 3	80	100	Hexane	800	10
5	Pest ICAL Mix 3	80	200	Hexane	800	20
6	Pest ICAL Mix 3	80	400	Hexane	800	40
7	Pest ICAL Mix 3	80	600	Hexane	800	60
8	Pest ICAL Mix 3	80	800	-	800	80

**Table 3: INTERMEDIATE PRIMARY STANDARD PREPARATION**

<b>PESTICIDES MIX 1: RESTEK</b>	<b>Stock Std. Conc. (µg/mL)</b>	<b>Final Conc. (µg/L)</b>	<b>Preparation</b>
Aldrin	8	80	Using a gas-tight syringe, measure 1000 µL of (Restek) Pesticides Mix 1 and 40 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
alpha-BHC	8	80	
beta-BHC	8	80	
delta-BHC	8	80	
gamma-BHC	8	80	
alpha-Chlordane	8	80	
gamma-Chlordane	8	80	
Heptachlor	8	80	
Heptachlor Epoxide	8	80	
Endosulfan I	8	80	
4,4'-DDD	16	160	
4,4'-DDE	16	160	
4,4'-DDT	16	160	
Dieldrin	16	160	
Endrin	16	160	
Endosulfan II	16	160	
Endosulfan Sulfate	16	160	
Endrin Aldehyde	16	160	
Endrin Ketone	16	160	
Methoxychlor	80	800	
<b>Surrogate: SUPELCO</b>			
Tetrachloro-m-xylene	200	80	
Decachlorobiphenyl	200	80	
<b>TOXAPHENE: AccuStandard</b>			
Toxaphene	1000	2000	Using a gas-tight syringe, measure 200 µL of (AccuStandard) Toxaphene standard and 50 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
<b>Surrogate: SUPELCO</b>			
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
<b>TECHNICAL CHLORDANE: AccuStandard</b>			
Technical Chlordane	1000	2000	Using a gas-tight syringe, measure 200 µL of (AccuStandard) Tech. Chlordane standard and 50 µL of (AccuStandard) Pest Surrogate and dilute with hexane to 100 mL.
<b>Surrogate: SUPELCO</b>			
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
<b>PESTICIDES MIX 3: SPEXCERTIPREP</b>			
2,4'-DDD	1000	80	Using a gas-tight syringe, measure 8 µL of (SpexCertiPrep) Pesticides Mix3 and dilute with hexane to 100 mL.
2,4'-DDE	1000	80	
2,4'-DDT	1000	80	
Oxychlordane	1000	80	
cis-Nanochlordane	1000	80	
trans-Nanochlordane	1000	80	
Mirex	1000	80	

**Table 4: INTERMEDIATE SECONDARY STANDARD PREPARATION**

<b>PESTICIDES MIX 1: SUPELCO</b>	Stock Std. Conc. (µg/mL)	Final Conc. (µg/L)	Preparation
Aldrin	2000	200	Using a gas-tight syringe, measure 10 µL of (Supelco) Pesticides Mix and 180 µL of (SpexCertiPrep) Methoxychlor and dilute with hexane to 100 mL.
alpha-BHC	2000	200	
beta-BHC	2000	200	
delta-BHC	2000	200	
gamma-BHC	2000	200	
alpha-Chlordane	2000	200	
gamma-Chlordane	2000	200	
Heptachlor	2000	200	
Heptachlor Epoxide	2000	200	
Endosulfan I	2000	200	
4,4'-DDD	2000	200	
4,4'-DDE	2000	200	
4,4'-DDT	2000	200	
Dieldrin	2000	200	
Endrin	2000	200	
Endosulfan II	2000	2002	
Endosulfan Sulfate	2000	200	
Endrin Aldehyde	2000	200	
Endrin Ketone	2000	200	
Methoxychlor	2000	2000	
Methoxychlor ( <i>SpexCertiPrep</i> )	1000		
Tetrachloro-m-xylene	200	200	
Decachlorobiphenyl	200	200	
<b>TOXAPHENE: <i>Ultra Scientific</i></b>			
Toxaphene	2500	2000	Using a gas-tight syringe, measure 20 µL of (Ultra Scientific) Toxaphene standard and 12.5 µL of (Supelco) Pest Surrogate and dilute with hexane to 25 mL.
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
<b>TECHNICAL CHLORDANE: <i>Ultra Scientific</i></b>			
Technical Chlordane	100	2000	Using a gas-tight syringe, measure 500 µL of (Ultra Scientific) Tech. Chlordane standard and 12.5 µL of (Supelco) Pest Surrogate and dilute with hexane to 100 mL.
Tetrachloro-m-xylene	200	100	
Decachlorobiphenyl	200	100	
<b>PESTICIDES MIX 3: <i>CPI</i></b>			
2,4'-DDD	1000	80	Using a gas-tight syringe, measure 4 µL of (CPI) Pesticides Mix3 and 40 µL of (AccuStandard) Mirex and dilute with hexane to 100 mL.
2,4'-DDE	1000	80	
2,4'-DDT	1000	80	
Oxychlordane	1000	80	
cis-Nanochlordane	100	80	
trans-Nanochlordane	100	80	
Mirex ( <i>AccuStandard</i> )	100	80	

**Note:** Table 2 and Table 2A may be interchanged. However, the source of LCS shall follow the source of the secondary standard or from a third vendor.

**Table 5: CHECK STANDARD PREPARATION (DCC/ICV)**

Standard	Compound Name	Intermediate Soln. Conc. (µg/L)	Source	Preparation			Final Conc. (µg/L)
				Aliquot (µL)	Dil.Soln./ Modifier	Final Vol. (µL)	
<b>PESTICIDES</b>							
DCC	Pest ICAL Mix	80/160/800	Restek	200	Hexane	800	20/40/200
	Surrogate Mix	80	AccuStandard				20
ICV	Pest ICV Mix	200/2000	Supelco / SpexCertiPrep	100	Hexane	1000	20/200
	Surrogate Mix	200	Supelco				20
<b>TOXAPHENE</b>							
DCC	Toxaphene ICAL	2000	AccuStandard	200	Hexane	800	500
	Surrogate Mix	100	AccuStandard				25
ICV	Toxaphene ICV	200	Ultra Scientific	200	Hexane	800	500
	Surrogate Mix	100	Supelco				25
<b>TECHNICAL CHLORDANE</b>							
DCC	Toxaphene ICAL	2000	AccuStandard	200	Hexane	800	500
	Surrogate Mix	100	AccuStandard				25
ICV	Toxaphene ICV	200	Ultra Scientific	200	Hexane	800	500
	Surrogate Mix	100	Supelco				25

**Table 6: SURROGATE STANDARD PREPARATION**

Compound Name	Stock Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/L)
			Aliquot (mL)	Dil.Soln./ Modifier	Final Vol. (mL)	
Surrogate Mix (Tetrachloro-m-xylene & Decachlorobiphenyl)	200	Supelco	2.0	Hexane	1,000	400

**Table 7: SPIKE STANDARD PREPARATION**

Compound Name	Stock/Intermediate Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/L)
			Aliquot (µL)	Dil.Soln./ Modifier	Dil. Vol. (mL)	
Pesticide Matrix Spike Mix	2,000	Supelco	20	Hexane	100	400/4000
Methoxychlor	1,000	SpexCertiPrep	360			

**Table 8: PERFORMANCE EVALUATION MIXTURE PREPARATION**

Compound Name	Stock/Intermediate Soln. Conc. (µg/mL)	Source	Preparation			Final Conc (µg/L)
			Aliquot (µL)	Dil.Soln./ Modifier	Dil. Vol. (mL)	
PEM (DDT + Endrin Mix)	500	Supelco	20	Hexane	100	100/100

**Table 9: CLP RETENTION TIME WINDOWS (CLP OLM4.2 D-79/PEST)**

Compound	Retention Time Window (minutes)	Compound	Retention Time Window (minutes)
alpha-BHC	± 0.05	Endrin Ketone	± 0.07
beta-BHC	± 0.05	4,4'-DDD	± 0.07
gamma-BHC(Lindane)	± 0.05	4,4'-DDE	± 0.07
delta-BHC	± 0.05	4,4'-DDT	± 0.07
Heptachlor	± 0.05	Endosulfan II	± 0.07
Aldrin	± 0.05	Endosulfan Sulfate	± 0.07
alpha-Chlordane	± 0.07	Methoxychlor	± 0.07
gamma-Chlordane	± 0.07	Aroclors	± 0.07
Heptachlor Epoxide	± 0.07	Toxaphene	± 0.07
Dieldrin	± 0.07		
Endrin	± 0.07	Tetrachloro-m-xylene	± 0.05
Endrin Aldehyde	± 0.07	Decachlorobiphenyl	± 0.10

**Table 10: ESTABLISHED LIMIT OF DETECTION (LOD) & LIMIT OF QUANTITATION (LOQ)**

<b>MATRIX: AQUEOUS</b>	<b>DL</b>	<b>LOD</b>	<b>LOQ</b>	<b>Unit</b>
Aldrin	0.005	0.01	0.1	µg/L
Alpha-BHC	0.005	0.01	0.1	µg/L
Beta-BHC	0.007	0.01	0.1	µg/L
Delta-BHC	0.007	0.01	0.1	µg/L
Gamma-BHC (Lindane)	0.005	0.01	0.1	µg/L
DDD (4,4)	0.005	0.01	0.1	µg/L
DDE (4,4)	0.005	0.01	0.1	µg/L
DDT (4,4)	0.005	0.01	0.1	µg/L
Dieldrin	0.005	0.01	0.1	µg/L
Endosulfan I	0.008	0.01	0.1	µg/L
Endosulfan II	0.005	0.01	0.1	µg/L
Endosulfan Sulfate	0.005	0.01	0.1	µg/L
Endrin	0.008	0.01	0.1	µg/L
Endrin Aldehyde	0.005	0.01	0.1	µg/L
Heptachlor	0.007	0.01	0.1	µg/L
Heptachlor epoxide	0.005	0.01	0.1	µg/L
Methoxychlor	0.05	0.1	1	µg/L
Alpha-Chlordane	0.005	0.01	0.1	µg/L
Gamma-Chlordane	0.005	0.01	0.1	µg/L
Endrin Ketone	0.005	0.01	0.1	µg/L
Toxaphene	0.25	0.5	2	µg/L
Technical Chlordane	0.005	0.25	1	µg/L
<b>SURROGATES</b>				
Tetrachloro-m-xylene	0.005	0.01	0.02	µg/L
Decachlorobiphenyl	0.005	0.01	0.02	µg/L

*Reference: Detection Limit Study dated April 20,2010.*

<b>PARAMETER (MATRIX: SOIL)</b>	<b>DL</b>	<b>LOD</b>	<b>LOQ</b>	<b>Unit</b>
Aldrin	0.02	0.4	2	µg/Kg
Alpha-BHC	0.02	0.4	2	µg/Kg
Beta-BHC	0.02	0.4	2	µg/Kg
Delta-BHC	0.27	0.4	2	µg/Kg
Gamma-BHC (Lindane)	0.2	0.4	2	µg/Kg
DDD (4,4)	0.02	0.4	2	µg/Kg
DDE (4,4)	0.02	0.4	2	µg/Kg
DDT (4,4)	0.02	0.4	2	µg/Kg
Dieldrin	0.02	0.4	2	µg/Kg
Endosulfan I	0.2	0.4	2	µg/Kg
Endosulfan II	0.2	0.4	2	µg/Kg
Endosulfan Sulfate	0.2	0.4	2	µg/Kg
Endrin	0.2	0.4	2	µg/Kg
Endrin Aldehyde	0.35	0.4	2	µg/Kg
Heptachlor	0.2	0.4	2	µg/Kg
Heptachlor epoxide	0.2	0.4	2	µg/Kg
Methoxychlor	2	4	10	µg/Kg
Alpha-Chlordane	0.2	0.4	2	µg/Kg
Gamma-Chlordane	0.2	0.4	2	µg/Kg
Endrin Ketone	0.2	0.4	2	µg/Kg
Toxaphene	5	10	50	µg/Kg
Technical Chlordane	10	20	50	µg/Kg
<b>SURROGATES</b>				
Tetrachloro-m-xylene	0.167	0.33	0.7	µg/Kg
Decachlorobiphenyl	0.167	0.33	0.7	µg/Kg

*Reference: Detection Limit Study dated April 5,2010.*

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURE**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
5 point Initial Calibration for analytes	Initially, as needed	RSD for all analytes ≤ 20% or Linear – least squares or inverse weighting factor: $r \geq 0.995$ .	Correct then problem then repeat initial calibration.		
Second –source Calibration Verification	Once per 5-point initial calibration	All analytes within ± 20of expected value from the ICAL.	Repeat injection of ICV. If the problem persists, perform troubleshooting and repeat the ICAL.		
Initial Calibration Verification Check	Daily, before sample analysis	All analytes within ± 20%	Correct the problem. If problem persists, repeat initial calibration		
Calibration Verification	Every 12 hours of analysis time and at the end of the analysis sequence	All analytes within ± 20%	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification		
Breakdown check (Endrin and DDT)	Every 12-hours	Degradation ≤ 15% of each analyte.	Repeat breakdown check		
Method Blank	One per preparation batch	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
LCS	One LCS per preparation batch	Within project QC limits	Re-prep and re-analyze the LCS and all associated samples		
Surrogate spike	Every sample, spiked sample, standard and method blank	Within project QC limits	Correct the problem then re-extract and re-analyze sample		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Within project QC limits	If chromatogram is indicative of matrix interference, discuss in case narrative. Otherwise, check for probable source of error and perform corrective action as necessary		
Confirmation	100% for all positive results	Same as primary column	If quantitation criteria are not met, use confirmation for qualitative identification only.		
Comments: 1. For flagging criteria refer to PSR. Otherwise, if MB is non-compliant, apply B to specific analyte(s) on all associated samples; apply J to all values between LOD and LOQ.			Reviewed By:		
			Date		

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: EPA 3520C / EPA 8081B**

Sample Prep SOP: EMAX-3520 Rev. 5  
Analytical SOP: EMAX-8081  
Conc Unit: µg/L  
Sample Amt(ml): 1000  
Extract Volume (mL): 10

Instrument ID: E8  
Extraction date: 9/12 & 9/16/13  
Extracted by: Iragasa / Jmuertigue  
Analysis date: 9/18 & 9/19/13  
Analyzed by: E. Santos

PARAMETER	CPI018WL	CPI018WC	CPI023WL	CPI023WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	RI18020A/B	RI18021A/B	RI18060A/B	RI18061A/B							
Aldrin	0.213	0.222	0.233	0.234	0.2	0.2	113	0.010	4.5	70 - 130	PASSED
alpha-BHC	0.219	0.231	0.250	0.245	0.2	0.2	118	0.014	5.9	70 - 140	PASSED
beta-BHC	0.241	0.275	0.299	0.269	0.2	0.3	135	0.024	8.7	70 - 140	PASSED
delta-BHC	0.235	0.248	0.263	0.261	0.2	0.3	126	0.013	5.3	70 - 140	PASSED
gamma-BHC (Lindane)	0.205	0.213	0.228	0.227	0.2	0.2	109	0.011	5.2	60 - 140	PASSED
DDD (4,4)	0.217	0.223	0.244	0.237	0.2	0.2	115	0.012	5.4	70 - 140	PASSED
DDE (4,4)	0.215	0.224	0.238	0.234	0.2	0.2	114	0.010	4.4	70 - 130	PASSED
DDT (4,4)	0.240	0.251	0.269	0.256	0.2	0.3	127	0.012	4.8	70 - 150	PASSED
Dieldrin	0.218	0.224	0.248	0.236	0.2	0.2	116	0.013	5.8	70 - 130	PASSED
Endosulfan I	0.206	0.213	0.228	0.224	0.2	0.2	109	0.010	4.7	70 - 130	PASSED
Endosulfan II	0.222	0.229	0.250	0.241	0.2	0.2	118	0.013	5.3	70 - 130	PASSED
Endosulfan sulfate	0.224	0.231	0.250	0.244	0.2	0.2	119	0.012	5.0	70 - 140	PASSED
Endrin	0.217	0.225	0.238	0.228	0.2	0.2	113	0.009	3.8	70 - 140	PASSED
Endrin Aldehyde	0.211	0.211	0.247	0.242	0.2	0.2	114	0.019	8.5	70 - 140	PASSED
Heptachlor	0.208	0.218	0.226	0.225	0.2	0.2	110	0.008	3.9	60 - 130	PASSED
Heptachlor epoxide	0.215	0.220	0.236	0.231	0.2	0.2	113	0.010	4.3	70 - 130	PASSED
Methoxychlor	2.160	2.253	2.462	2.477	2	2	117	0.157	6.7	70 - 140	PASSED
alpha-Chlordane	0.211	0.219	0.232	0.228	0.2	0.2	111	0.009	4.2	70 - 130	PASSED
gamma-Chlordane	0.224	0.232	0.248	0.244	0.2	0.2	119	0.011	4.6	70 - 140	PASSED
Endrin Ketone	0.223	0.221	0.240	0.236	0.2	0.2	115	0.009	4.1	70 - 130	PASSED
Tcx	0.365	0.396	0.398	0.406	0.4	0.4	98	0.018	4.6	30 - 130	PASSED
DCB	0.356	0.368	0.368	0.367	0.4	0.4	91	0.006	1.6	60 - 130	PASSED

**Appendix 2: DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: EPA 3550C / EPA 8081B**

Sample Prep SOP: EMAX-3550 Rev. 3  
Analytical SOP: EMAX-8081 Rev. 7  
Conc Unit: µg/Kg  
Sample Amt(g): 30  
Extract Volume (mL): 10

Instrument ID: E8  
Extraction date: 1/16 & 1/21/14  
Extracted by: J. Villena  
Analysis date: 1/29 & 1/31/14  
Analyzed by: E. Santos

PARAMETER	CPA019SL	CPA019SC	CPA033SL	CPA022SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	MA29007A	MA29008A	MA31013A	MA31014A							
Aldrin	6.30	6.29	7.15	7.07	6.67	6.7	101	0.474	7.1	60 - 130	PASSED
alpha-BHC	6.21	6.20	7.26	7.29	6.67	6.7	101	0.616	9.1	50 - 140	PASSED
beta-BHC	6.28	6.31	7.47	7.44	6.67	6.9	103	0.670	9.7	60 - 140	PASSED
delta-BHC	6.59	6.63	7.59	7.53	6.67	7.1	106	0.546	7.7	60 - 140	PASSED
gamma-BHC (Lindane)	6.29	6.27	7.08	6.96	6.67	6.6	100	0.431	6.5	60 - 130	PASSED
DDD (4,4)	7.53	7.63	7.47	7.42	6.67	7.5	113	0.090	1.2	70 - 140	PASSED
DDE (4,4)	6.67	6.75	7.47	7.39	6.67	7.1	106	0.419	5.9	60 - 140	PASSED
DDT (4,4)	8.07	8.16	8.82	8.89	6.67	8.5	127	0.431	5.1	70 - 150	PASSED
Dieldrin	6.56	6.58	7.45	7.40	6.67	7.0	105	0.493	7.0	60 - 140	PASSED
Endosulfan I	6.14	6.12	7.23	7.11	6.67	6.6	100	0.605	9.1	50 - 130	PASSED
Endosulfan II	6.74	7.00	7.58	7.53	6.67	7.2	108	0.410	5.7	70 - 130	PASSED
Endosulfan sulfate	7.02	7.31	7.79	7.74	6.67	7.5	112	0.365	4.9	60 - 140	PASSED
Endrin	7.16	7.29	7.43	7.34	6.67	7.3	110	0.112	1.5	70 - 140	PASSED
Endrin Aldehyde	6.53	6.68	7.37	7.17	6.67	6.9	104	0.396	5.7	60 - 140	PASSED
Heptachlor	6.19	6.28	7.08	7.14	6.67	6.7	100	0.506	7.6	60 - 130	PASSED
Heptachlor epoxide	6.32	6.59	7.29	7.31	6.67	6.9	103	0.502	7.3	60 - 130	PASSED
Methoxychlor	81.5	84.7	92.7	92.8	66.7	88	132	5.686	6.5	60 - 140	PASSED
alpha-Chlordane	6.59	6.60	7.33	7.28	6.67	6.9	104	0.410	5.9	60 - 130	PASSED
gamma-Chlordane	6.52	6.64	7.41	7.32	6.67	7.0	105	0.458	6.6	60 - 130	PASSED
Endrin Ketone	6.61	6.83	7.52	7.52	6.67	7.1	107	0.471	6.6	70 - 130	PASSED
Tcx	10.71	10.24	11.91	11.83	13.3	11.2	84	0.827	7.4	50 - 140	PASSED
DCB	13.1	12.7	13.9	13.7	13.3	13.4	100	0.545	4.1	50 - 140	PASSED





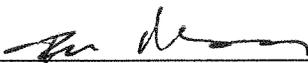


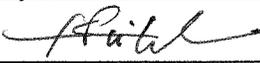
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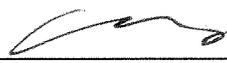
## STANDARD OPERATING PROCEDURES

**POLYCHLORINATED BIPHENYLS (PCB) AND POLYCHLORINATED TERPHENYLS (PCT) BY GAS CHROMATOGRAPHY**

SOP No.: EMAX-8082 Revision No. 5 Effective Date: 02-Jul-14

Prepared By: Tu Nisamaneepong  Date: 07-02-14

Approved By: Kenette Pimentel   
QA Manager Date: 07-02-14

Approved By: Caspar Pang   
Laboratory Director Date: 07-02-14

Control Number: 8082-05-

**1.0 SCOPE AND APPLICATION**

- 1.1. This procedure is used to determine the concentration of Polychlorinated Biphenyls (PCBs) and Polychlorinated Terphenyls (PCTs) as Aroclors in soil, sediment, sludge, and wastewater samples by gas chromatography method.
- 1.2. This SOP is an adaptation of EPA 8082A. Since EPA 8082A is an update and enhancement of 8082, this SOP is also applicable to EPA 8082.

**2.0 SUMMARY OF METHOD**

- 2.1. This method provides gas chromatographic conditions for the detection of PCB and PCT compounds with dual Electron Capture Detector (ECD). The samples are extracted in methylene chloride, exchanged to hexane and cleaned up by appropriate method before GC analysis.
- 2.2. **Interferences**
  - 2.2.1. Interferences by phthalate esters co-extracted from the sample or introduced during sample preparation can pose a major problem in PCB and PCT determinations. Interferences can be minimized by avoiding contact with any plastic materials and checking all solvents for phthalate contamination. Glassware must be scrupulously cleaned. Sulfuric acid/permanganate cleanup technique can be used for Phthalate esters removal from the extract.
  - 2.2.2. The presence of elemental sulfur will result in broad peaks that may cause chromatographic interfere with the determination of PCBs and PCTs. Sulfur contamination is most likely present in sediment samples. The TBA procedure, GPC or other cleanup technique can be used for sulfur removal from the extract.
  - 2.2.3. Other interferences such as aliphatic compounds, aromatics and nitrogen-containing compounds may be eliminated by using Florisil cleanup.

**3.0 DETECTION LIMITS**

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**
  - 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
  - 3.1.2. Refer to Table 6 for established DL, LOD and LOQ values.

## STANDARD OPERATING PROCEDURES

**POLYCHLORINATED BIPHENYLS (PCB) AND POLYCHLORINATED TERPHENYLS (PCT) BY GAS CHROMATOGRAPHY**SOP No.: EMAX-8082 Revision No. 5 Effective Date: 02-Jul-14**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and shall require dilution to properly quantitate.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.
- 4.3. Typical dynamic ranges are:

	PCBs	PCTs
Water ( $\mu\text{g/L}$ )	0.5 - 20.0	2.0 - 10.0
Soil ( $\mu\text{g/Kg}$ )	16.67 – 666.67	40 - 500

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1. Holding Time**

- 5.1.1. PCBs and PCTs are very stable in a variety of matrices and holding times may be as long as a year.
- 5.1.2. Analysis should be within 40 days after extraction completion date.

**5.2. Preservation**

- 5.2.1. Samples and extract should be kept at  $\leq 6^{\circ}\text{C}$ .

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA014 Detection Limit
- 6.3. EMAX-QA08 Corrective Action
- 6.4. EMAX-QC02 Analytical Standard Preparation
- 6.5. EMAX-SM04 Analytical and QC Labeling
- 6.6. EMAX-3520 Extraction, Continuous Liquid/Liquid
- 6.7. EMAX-3546 Extraction, Microwave
- 6.8. EMAX-3550 Extraction, Pulse Sonication
- 6.9. EMAX-3540 Extraction, Soxhlet
- 6.10. EMAX-3580 Waste Dilution
- 6.11. EMAX-3620 Cleanup, Florisil

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- 6.12. EMAX-3660 Cleanup, Sulfur
- 6.13. EMAX-3665 Cleanup, Acid/Permanganate

**7.0 SAFETY**

- 7.1. Read all SDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. ECD contains minute quantity of Radioactive Ni (63), a wipe test performed by experienced personnel or manufacturer should be conducted semiannually or sooner if potential problem is suspected. Observe standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of the body, rinse the affected body part thoroughly with copious amounts of water. If irritations or any other discomfort related to the incident persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS & REAGENTS****8.1. Instruments and Supplies**

Gas Chromatography	HP 5890 Series II
Detector	Dual Electron Capture Detectors
Column	RTX CLPESTI (30 m x 0.32 mm x 0.5 µm) RTX CLPESTII (30 m x 0.32 mm x 0.25 µm) (Alternate columns may be used after verification of performance)
Data System	EZ Chrom
Auto Sampler	HP Model 7673B or equivalent
Gas	Ultra-high purity hydrogen Ultra-high purity nitrogen
Microsyringes	10, 25, 100 and 500 µL with a 0.006 mm ID needle
Volumetric Flasks	0,50, and 100 ml with ground glass stopper
Transfer Pipette	Pasteur

**8.2. Chemicals and Reagents**

Solvent (GC-grade)	Pesticide-free grade hexane, methylene chloride, isopropanol
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**9.0 STANDARDS****9.1. Preparation**

## STANDARD OPERATING PROCEDURES

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9.1.1. Prepare and label analytical standards according to EMAX-QC02 and EMAX-SM04.

9.2. **Stock Standard**

9.2.1. Purchase Primary Calibration stock standards as certified solutions at 1000 mg/L. After opening, transfer the stock standard to an inert vial and store with a minimum headspace.

9.2.2. Purchase a Secondary set of stock standards from a different source to verify the concentration of the first set of standards. Treat the secondary standards similarly as the primary standards.

9.2.3. Purchase LCS/MS and surrogate standards as certified solutions at 1000 mg/L and 200 mg/L, respectively.

9.2.4. Store all standards at  $\leq 6^{\circ}\text{C}$ , unless otherwise specified.

9.3. **Intermediate Standard**

9.3.1. Prepare intermediate standards (for both primary and secondary) at 4000  $\mu\text{g/L}$  as suggested in Table 1.

9.4. **Initial Calibration Standard (ICAL)**

9.4.1. Prepare a minimum of five calibration standards using primary intermediate standard according to Table 2.

9.5. **Initial Calibration Verification (ICV)**

9.5.1. Prepare ICV using the secondary intermediate standard as described in Table 3.

9.6. **Daily Calibration Check Standard (DCC)**

9.6.1. Prepare DCC using the primary intermediate standard as described in Table 3.

9.7. **Surrogate Standard**

9.7.1. Prepare surrogate standard as described in Table 4.

9.8. **LCS/MS Spike Standard**

9.8.1. Prepare LCS/MS spike standard as described in Table 4.

**10.0 PROCEDURES**

10.1. **Sample Preparation**

10.1.1. Aqueous samples shall be prepared as described in EMAX-3520.

10.1.2. Solid samples shall be prepared as described in EMAX-3550 or EMAX-3546. If necessary, clean up with sulfuric acid as described in EMAX-3665 is performed.

10.2. **Instrument Parameters**

10.2.1. EPA 8082A requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 1 to 5  $\mu\text{L}$ .

10.2.2. Gas Pressure

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Nitrogen Pressure : 40 psi

Hydrogen Pressure : 50 psi

10.2.3. Temperature Program

<b>RESTEK RTX CLPest 1 &amp; 2</b>	
Initial Temp	130°C, hold for 0.5 minute
Rate 1	25°C/min
Temp 1	230°C, hold for 2 minutes
Rate 2	5°C/min
Temp 2	260°C, hold for 0.2 minute
Rate 3	25°C/min
Final Temp	290°C, hold for 1 minute (PCB) 290°C, hold for 17 mins. (PCB+PCT)
Injector	230°C
Detector	300°C
Injection Volume	1 µL

10.3. **Calibration**10.3.1. Initial Calibration (ICAL)

- 10.3.1.1. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclor 1016 and 1260 at a minimum of five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors.
- 10.3.1.2. A separate set of calibration standards containing Aroclor 5460 is also prepared.
- 10.3.1.3. Initial calibration standards (or as suggested in Table 2) of Aroclors 1016, 1260, and 5460 are analyzed by direct injection.
- 10.3.1.4. Three to five characteristic peaks from each Aroclor are chosen for quantitation. Peaks should be at least 25% of the height of the largest Aroclor peak. Tabulate each peak area response against concentration of injected standard. Calculate each calibration factor according to Eq. 10.6.1.1. A minimum of five sets of calibration factors will be generated for each Aroclor.
- 10.3.1.5. Application of ICAL Curve for Quantitation
- 10.3.1.5.1. Generate a summary of calibration factors for each analyte at each concentration using Eq-10.6.1. Calculate the Average Calibration

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Factor (ACF), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to, Eq. 10.6.2, Eq. 10.6.3 and Eq. 10.6.4, respectively.

- If RSD is  $\leq 20\%$ , ACF may be applied.
- Apply Inverse Weighting Factor ( $1/y$  or  $1/y^2$ ;  $y$  being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.995$ .
- Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.995$ .
- It may be appropriate to force the regression through zero for specific analytes<sup>1</sup>. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.1.6. In situations where a particular Aroclor is of interest for a specific project, a minimum five-point calibration curve of that Aroclor may be employed.

10.3.1.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.1.8. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.2. Initial Calibration Verification (ICV)

10.3.2.1. Prepare a mid-level calibration standard of Aroclors 1260 + 1016 and Aroclor 5460 from a second source. Analyze these after the initial calibration to verify the ICAL.

10.3.2.2. Calculate the %Difference (%D) using Eq. 10.6.2.1. This should be equal to or less than 20%.

10.3.2.3. The standards of the other Aroclors are used to determine a single-point calibration factor for each Aroclor and pattern recognition. It can be analyzed before or after the Aroclor 1016/1260 and 5460 standards.

10.3.3. Daily Calibration Check (DCC)

<sup>1</sup> SW846 Method 8000B, Section 7.5.3; SW846 Method 8000C, Section 11.5.2.1

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- 10.3.3.1. For daily run, analyze DCC (mid-level Aroclor 1016 + 1260 and 5460 standards) at the start of the 12-hour shift prior to sample analysis, and close the analytical run with an ending DCC. A 12-hour shift interval must not exceed 20 samples.
  - 10.3.3.2. The calibration check process does not require analysis of the other Aroclor standards used for pattern recognition.
  - 10.3.3.3. Calculate the %D by using Eq. 10.6.2.1. Refer to Appendix 1 for acceptance criteria.
  - 10.3.3.4. For projects wherein specific PCB is expected to be found, analyze DCC of that particular PCB at 500 µg/L.

**10.4. Analysis****10.4.1. Analytical Sequence**

10.4.1.1. Following the instrument data acquisition software, prepare the analytical sequence file as suggested below:

- Instrument Blank
- Minimum Five Calibration Standards of Aroclors 1016/1260
- Minimum Five Calibration Standards of Aroclor 5460
- Calibration Standards of Aroclor 1254, 1248, 1242, 1232, 1221, 1262, 5432 and 5442 (as necessary) \*
- Initial Calibration Verification of Aroclor 1016/1260 and Aroclor 5460
- Method Blank
- Lab Control Sample
- Samples (up to 12 hours but not more than 20 samples)
- Continuing Calibration Standard of of Aroclor 1016/1260 and Aroclor 5460 or other calibrated Aroclors

**10.4.2. Sample Analysis**

- 10.4.2.1. Transfer a minimum of 0.5 mL of extract to a 2-mL autosampler vial (or equivalent) using a Pasteur pipette. Cap the vial with a teflon septum and seal with an aluminum rim.
- 10.4.2.2. Introduce extract into the gas chromatograph using direct injection technique (1 to 5 µl) after all quality control criteria have been met.
- 10.4.2.3. If the response exceeds the linear range of the system, dilute the sample and re-analyze.

**10.4.3. Sample Result Evaluation**

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\* Include Calibration standards for Aroclor 1254, 1248, 1242, 1232, 1221, 1262, 1268, 5432 and 5442 if pattern is found in the sample.

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- 10.4.3.1. Check QC parameters as soon as the data is available.
- ✓ Check LCS recovery that it is within QC limits (Appendix 1).
  - ✓ Check MB that is project compliant.
  - ✓ Check retention time.
  - ✓ Check surrogate recoveries against Appendix 1.
  - ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
  - ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, order re-extraction for the affected sample(s).
- 10.4.3.2. Identification of a multicomponent analyte in the sample is based on pattern recognition in conjunction with the elution of three to five peaks on both GC columns.
- 10.4.3.3. Positive identification is made when a target peak falls within the retention time window on both columns established by the standard reference compound.
- 10.4.3.3.1. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:
- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW<sup>2</sup> whichever is greater.
  - ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.
- 10.4.3.4. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Eq. - 10.6.4.2.
- If the RPD is less than 40% and the pattern peaks do not indicate any anomalies, report the higher result.
  - If the RPD is less than 40% and the pattern peaks indicate an anomaly, report the result from the better pattern peaks.
  - If the RPD is greater than 40%, use professional judgment. If no anomaly is found, report the higher result.

10.4.4. Retention Time Window10.4.4.1. **Establishing RTW**<sup>2</sup> CLP-OLM4.2 Table 1 D-79/PEST

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- 10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.
- 10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each of the major peaks used for calibration.
- 10.4.4.1.3. Determine the width of RTW by  $\pm 3X$  SD.
- 10.4.4.2. **Evaluating RTW**
- 10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained.
- 10.4.4.2.2. For new instruments, in the interim use the CLP retention time window for Aroclors ( $\pm 0.07$  min) until RTW is obtained for the new instrument parameters condition.
- 10.4.4.3. **Application of RTW**
- 10.4.4.3.1. Establish the center of absolute retention time for each of the characteristic peak to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.
- 10.4.4.3.2. Whenever the observe retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.
- 10.4.4.4. **Updating RTW**
- 10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:
- Yearly RTW update
  - Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
  - Major instrument maintenance (e.g. replacements of detector or column; temperature program change, etc.)
- 10.4.5. **Manual Integration**
- 10.4.5.1. Refer to EMAX-DM01 for details of manual integration.
- 10.4.6. **Dealing with Carryover**
- 10.4.6.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- 10.4.6.2. If there was no target analyte detected (as found in the sample that exceeded calibration range), proceed with data reduction.
- 10.4.6.3. If there was a target analyte detected (as found in the sample that exceeded the calibration range), re-analyze the sample to rule-out carry-over. If carry-over is confirmed, proceed with data reduction and report the data from the re-analysis.

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- 10.5.1. Make a copy of the analytical run log and highlight the data to be reported.
- 10.5.2. Collate the reportable raw data. Segregate raw data for samples, QC data and calibration data using section dividers.
- 10.5.3. Keep all other unused data generated during the analysis in the analytical folder and mark with "For record only".
- 10.5.4. Proceed to report generation.

**10.6. Calculations****10.6.1. Initial Calibration****10.6.1.1. Calculate for Calibration Factor (CF)**

$$CF = \frac{R_a}{C_a} \quad \text{Eq.-10.6.1.1}$$

*where:*

- $R_a$  - response for analyte measured in peak area
- $C_a$  - Concentration of analyte ( $\mu\text{g/L}$ )  $\times 1/N$
- $N$  - number of identification peak in each Aroclor

**10.6.1.2. Calculate for Average Calibration Factor (ACF)**

$$ACF = \frac{\sum CF_a}{n} \quad \text{Eq.-10.6.1.2}$$

*where:*

- ACF - average calibration factor
- $\sum CF_a$  - sum of calibration factors
- $n$  - number of calibration points

**10.6.1.3. Calculate for Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}} \quad \text{Eq.-10.6.1.3}$$

*where:*

- SD - standard deviation
- $x_i$  - result at  $i^{\text{th}}$  measurement
- $\bar{x}$  - mean of all  $x$  measurements

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N - number of measurements

10.6.1.4. **Calculate for % Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{ACF} * 100\% \quad \text{Eq.-10.6.1.4}$$

where:

RSD - relative standard deviation

SD - standard deviation

ACF - average calibration factor

10.6.1.5. **Calculate for Least Square Line Regression**

$$y = ax + b \quad \text{Eq.-10.6.1.5}$$

where:

y – Response Factor

x – Concentration

a = x1 = slope of the line

$$a = \frac{\sum(x - \bar{x})(y - \bar{y})}{(x - \bar{x})^2}$$

 $\bar{x}$  - Average of amount ratios $\bar{y}$  - Average of response ratios

b = x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

10.6.1.6. **Calculate for Inverse Weighting Factor**

$$y = ax + b \quad \text{Eq.-10.6.1.6}$$

where:

y – Response Factor

x – Concentration

a – x1 = slope of the line

$$a = \frac{\sum[(x - x_a)(y - y_a)]}{\sum(x - x_a)^2}$$

where:

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$$x_a = \Sigma[x(1/x)/\Sigma(1/x)]$$

$$y_a = \Sigma[y(1/x)/\Sigma(1/x)]$$

$$x_a = \Sigma[x(1/x^2)/\Sigma(1/x^2)]$$

$$y_a = \Sigma[y(1/x^2)/\Sigma(1/x^2)]$$

$b = x_0 =$  intercept of the line

$$b = y_a - a * x_a$$

10.6.2. Calibration Check/Continuing Calibration10.6.2.1. **Calculate Percent Difference**

$$\% D = \frac{ACF - CF}{ACF} * 100\% \quad \text{Eq.-10.6.2.1}$$

where:

ACF - average calibration factor

CF - calibration factor at calibration check standard

10.6.3. Sample Results

10.6.3.1. Identify 3 to 5 major peaks as quantitative peaks for each Aroclor. Establish the ACF of each peak as outlined in Section 10.6.1.1. Calculate the concentration of each peak in Aroclor according to Eq. - 10.6.3.2 or Eq. - 10.6.3.3. The concentration of Aroclor equals the sum of the concentrations of these major peaks according to Eq. - 10.6.3.4.

10.6.3.2. **Water Samples**

$$C_n = \left( \frac{R_a}{ACF} \right) \left( \frac{V_e}{S_a} \right) DF \quad \text{Eq-10.6.3.2}$$

where:

$C_n$  - Concentration of characteristic peak n measured ( $\mu\text{g/L}$ )

$R_a$  - Total response of analyte in peak area

ACF - Average calibration factor

$V_e$  - Volume of extract in ml

$S_a$  - Sample amount in ml

DF - Dilution factor of sample extract

10.6.3.3. **Soil Samples**

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$$C_n = \left( \frac{R_a}{ACF} \right) \left( \frac{V_e}{S_a (\% \text{ Solid})} \right) DF \quad \text{Eq.10.6.3.3}$$

where

- $C_n$  - Concentration of characteristic peak n measured ( $\mu\text{g}/\text{kg}$ )  
 $R_a$  - Total response of analyte in peak area  
ACF - Average calibration factor  
 $V_e$  - Volume of extract in ml  
 $S_a$  - Sample Amount in g  
% Solid -  $\frac{100 - \% \text{ moisture}}{100}$   
DF - Dilution factor of the sample extract

10.6.3.4. **Final Aroclor Concentration**

$$C = C_1 + C_2 + \dots + C_n \quad \text{Eq.-10.6.3.4}$$

where:

- $C$  - Concentration of Aroclor in sample ( $\mu\text{g}/\text{L}$  or  $\mu\text{g}/\text{kg}$ )  
 $C_1$  - Concentration of characteristic peak 1  
 $C_2$  - Concentration of characteristic peak 2  
n - Characteristic peak number in each Aroclor

10.6.4. Accuracy and Precision10.6.4.1. **Percent Recovery**

$$\% R = \frac{C_f - C}{C_s} * 100 \quad \text{Eq.-10.6.4.1}$$

where:

- %R - percent recovery  
 $C_f$  - concentration of spiked sample  
 $C_s$  - concentration of spike  
C - concentration of unspiked sample (For LCS recovery, C=0)

10.6.4.2. **Relative Percent Difference**

$$\% RPD = \frac{|C_1 - C_2|}{\left( \frac{C_1 + C_2}{2} \right)} \times 100 \quad \text{Eq.-10.6.4.2}$$

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where:

- %RPD - Relative Percent Difference
- C<sub>1</sub> - Measured concentration of the first sample aliquot
- C<sub>2</sub> - Measured concentration of the second sample aliquot

**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WDB1C .exe
- 10.7.2. Generate Lab Chronicle using LABCHRN1.exe
- 10.7.3. Generate sample results using F1NV3C .exe
- 10.7.4. Generate the QC Summary file using QCV3CN .exe
- 10.7.5. Generate the case narrative using CN1.exe.
- 10.7.6. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analysis.
  - 10.7.6.1. Case Narrative
  - 10.7.6.2. Lab Chronicle
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  - 10.7.6.8. DCC Summary
  - 10.7.6.9. Analytical Run Log
  - 10.7.6.10. Sample Preparation Log
  - 10.7.6.11. Non-Conformance Report (if any)

**10.8. Data Review**

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the project specific requirements (PSR).
  - 10.8.1.1. If any of the checkpoints below indicate a problem, re-analysis is required.
    - ✓ Check that all samples required for analysis are analyzed.
    - ✓ Check that samples are extracted and analyzed within holding time.
    - ✓ Check that all calibration requirements are fulfilled.
    - ✓ Check the chromatogram of all positively identified Aroclor patterns that they are qualitatively identified and qualitatively accurate.
    - ✓ Check surrogate recoveries against required limits.

## STANDARD OPERATING PROCEDURES

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✓ Check that concentration of target analytes are within calibration range.

10.8.2. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.3. Submit the analysis package for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Refer to Form 8082FM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back in control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

<b>Maintenance Activity</b>	<b>Description</b>	<b>Frequency</b>
Autosampler Check	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check liner as necessary. Check instrument performance (e.g., daily calibration check, instrument blank, DDT/Endrin breakdown).	Daily prior to analysis
Documentation	Record maintenance in instrument service logs.	Daily prior to analysis
Leak Test	Perform inlet pressure decay test.	Every 6 months or as necessary
System Cleaning	Remove dust from fans and vent covers, inspect and clean inlet and detector where applicable.	Every 6 months or as necessary
Check Flow Path Components	Check and replace the following as necessary: tubing assembly, union, sample probe, and loop.	Once a year or as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting.	Once a year

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**11.0 QUALITY CONTROL****11.1. Preparative Batch**

- 11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD and a maximum of 20 field samples of similar matrix.
- 11.1.2. In the absence of MS/MSD, LCS/LCD is prepared.
- 11.1.3. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.
- 11.1.4. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within  $\pm 20\%$  of the expected value.

**11.2. Analytical Batch QC**

- 11.2.1. Instrument Blank must be analyzed daily to establish zero baseline or background value.
- 11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.2 and Appendix 1.

**11.3. Method QC**

- 11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.
- 11.3.2. A valid LOD and LOQ must exist prior to sample analysis.
- 11.3.3. A valid ICAL must exist prior to sample analysis.
- 11.3.4. Instrument performance must be checked prior to sample analysis. Check Appendix 1 for acceptance criteria.
- 11.3.5. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

**12.0 CORRECTIVE ACTION**

- 12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.
- 12.2. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.
  - 12.2.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.
  - 12.2.3. If the highest ICAL point appears to be saturated, drop the highest point.
  - 12.2.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

*Note: The lowest calibration point identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects to which the ICAL will be used.*

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- 12.2.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
  - 12.2.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
  - 12.2.5.2. Check the gas flow
  - 12.2.5.3. Prepare a fresh standard and repeat calibration
- 12.2.6. If the problem persists, inform the supervisor.
- 12.3. If the ICV is non-compliant, consider the following suggestions to correct the problem:
  - 12.3.1. Re-analyze ICV ( to rule out poor injection)
  - 12.3.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error
  - 12.3.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration
  - 12.3.4. If the problem persists, inform the supervisor
- 12.4. If the instrument blank is non-compliant, consider the following suggestions to correct problem:
  - 12.4.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new auto sampler syringe.
  - 12.4.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
  - 12.4.3. Rule out preparation contamination by preparing a new instrument blank
  - 12.4.4. If the problem persists, inform the supervisor.
- 12.5. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:
  - 12.5.1. Change the liner
  - 12.5.2. Clean injection port
  - 12.5.3. Prepare new standard
  - 12.5.4. Cut or replace column
  - 12.5.5. Clean the detector
  - 12.5.6. Rule out leaks by checking all connections
  - 12.5.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL
- 12.6. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
  - 12.6.1. Rule out instrument contamination by checking instrument blank
  - 12.6.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01
  - 12.6.3. Rule out glassware contamination used for extraction as described in EMAX-QC07
  - 12.6.4. Re-extract MB and the associated samples with reagents free of contamination or with newly

## STANDARD OPERATING PROCEDURES

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opened reagents

12.6.5. If the problem persists, inform the supervisor

12.7. If LCS is non-compliant, perform the following suggestions to correct the problem:

12.7.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level

12.7.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.

12.7.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.

12.8. Initiate a Non-Conformance Report (NCR) when the following circumstances occur:

- Anomalies other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

12.8.1. Refer to EMAX-QA08 for NCR details.

12.9. For other problems encountered, inform the supervisor immediately for further instructions.

**13.0 POLLUTION PREVENTION**

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standard s in fume hoods.

**14.0 WASTE MANAGEMENT**

14.1. No samples shall be dumped on the laboratory sink.

14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.

14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.

14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.

15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

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- 15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.3. **Detection Limit (DL)** – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.1.4. **Limit of Detection (LOD)** – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1%.
- 15.1.5. **Limit of Quantitation (LOQ)** – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.1.6. **Safety Data Sheet (SDS)** – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.1.7. **Calibration** – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8. **Calibration Blank** – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.9. **Characteristic Peaks** – are major identifying and quantifying peaks for each type of Aroclor.
- 15.1.10. **Instrument Method** – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.11. **Method Blank** – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. **Lab Control Sample (LCS)** – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.13. **Lab Control Sample Duplicate (LCSD)** – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.14. **Sample** – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.15. **Sample Duplicate** – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.16. **Sub-sample** – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

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- 15.1.17. Matrix – is a component or form of a sample.
- 15.1.18. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.19. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.20. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.
- 15.1.21. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.
- 15.1.22. Reagent Soil – organic-free Ottawa sand or equivalent.

**15.2. Application of QC Procedures**

- 15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing PCB analysis by GC/MS. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

**15.3. Department of Defense (DoD) and Department of Energy (DoE) Projects**

- 15.3.1. Samples from DoD and DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.

**16.0 REFERENCES**

- 16.1. "Test Methods for Evaluating Solid Waste, Physical and Chemical Methods" (SW846) Method 8082A Rev. 1, Feb. 2007 and Method 8082 Rev. 0, Dec. 1996
- 16.2. "Test Methods for Evaluating Solid Waste, Physical and Chemical Methods" (SW846) Method 8000B, Rev. 2 Dec. 1996 and Method 8000C Rev. 3, March 2003
- 16.3. EMAX Quality Systems Manual, as updated

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## STANDARD OPERATING PROCEDURES

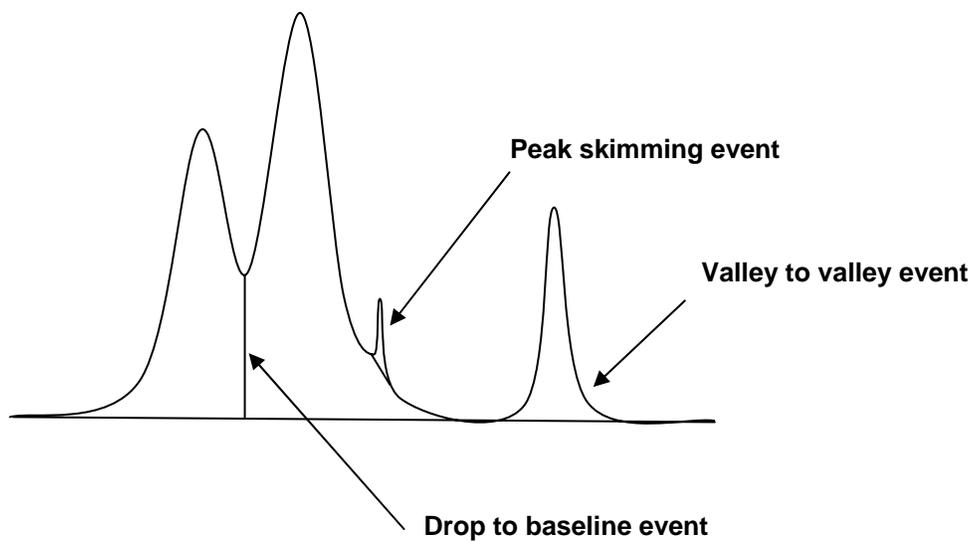
**POLYCHLORINATED BIPHENYLS (PCB) AND POLYCHLORINATED TERPHENYLS (PCT) BY GAS CHROMATOGRAPHY**SOP No.: EMAX-8082 Revision No. 5 Effective Date: 02-Jul-14

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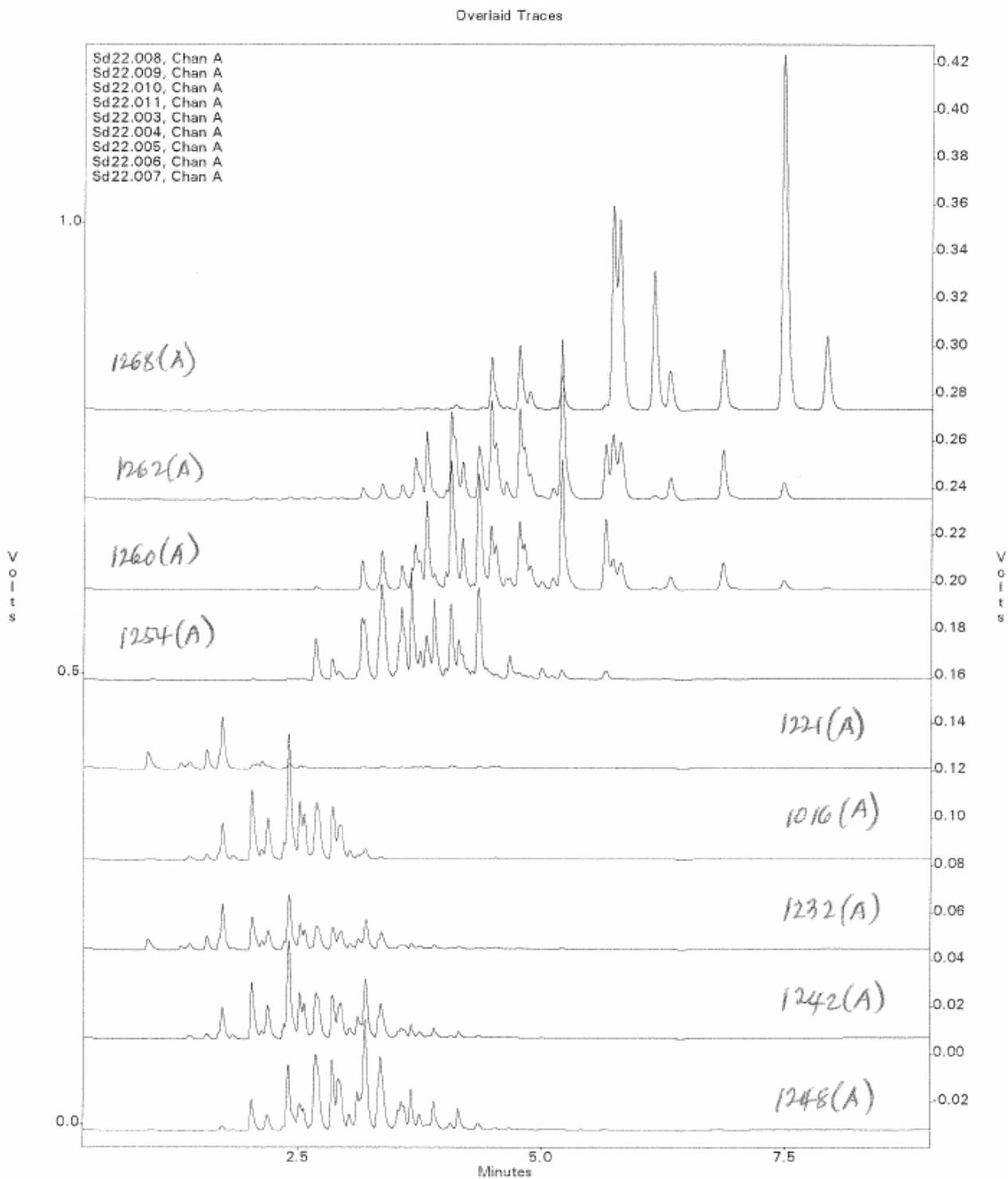
- 17.1.7. Figure 5 Typical Retention Time Window Summary
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**Figure 1: PEAK EVALUATION TECHNIQUE**

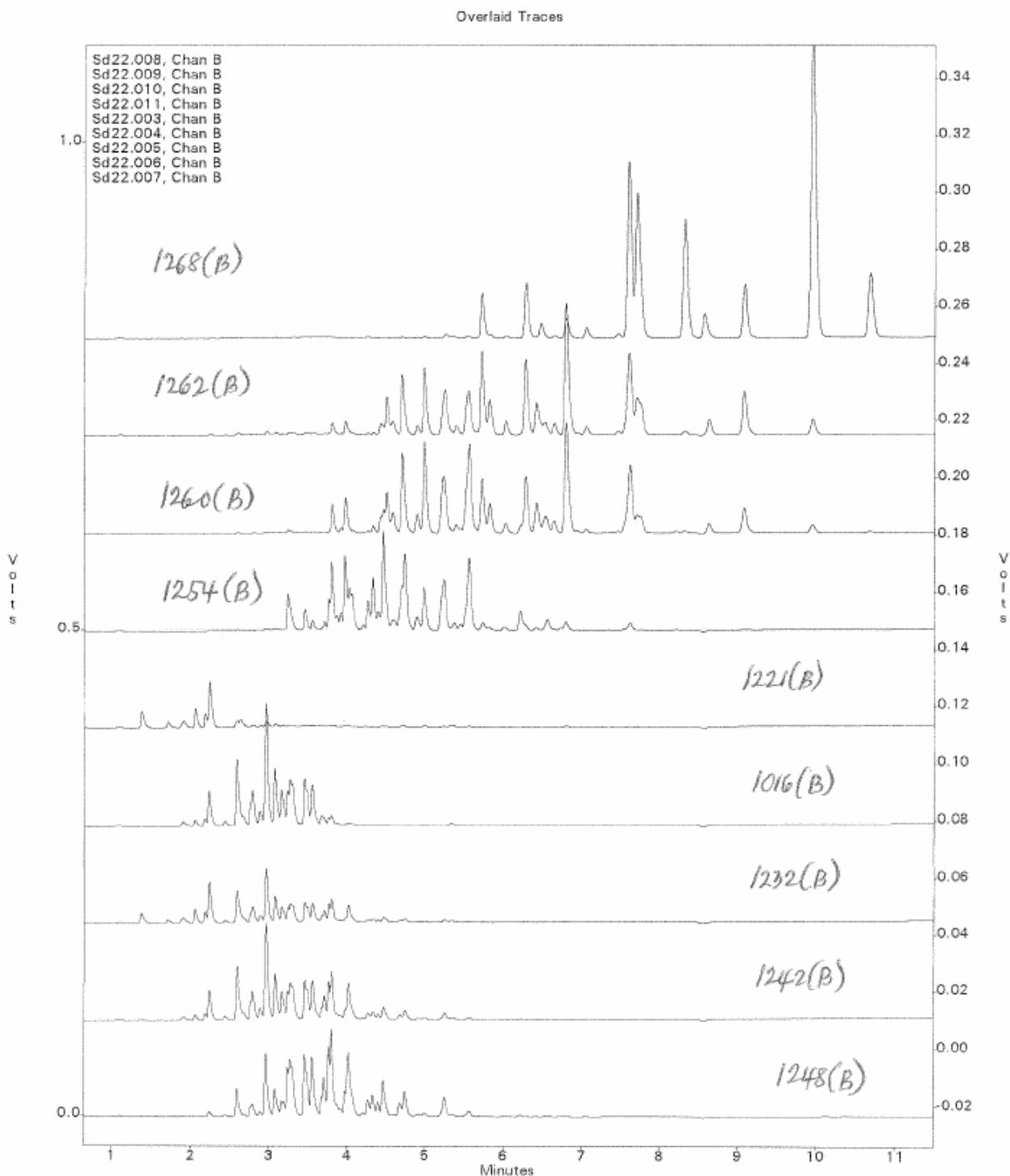
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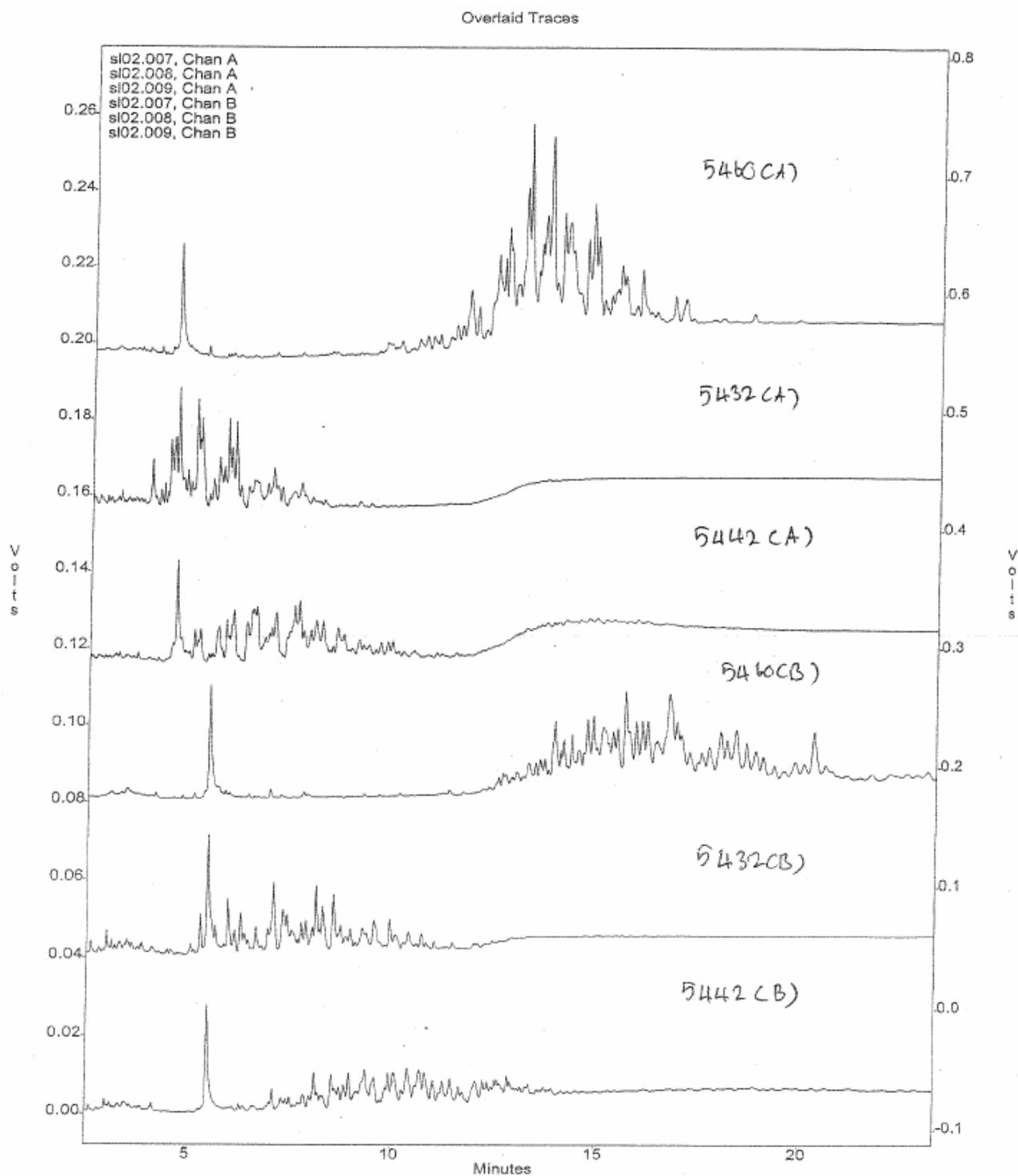
**Figure 2A: TYPICAL PCB PATTERNS**



**Figure 2A: TYPICAL PCB PATTERNS**



**Figure 2B: TYPICAL PCT PATTERNS**



**Figure 3A: TYPICAL 1016 / 1260 CHROMATOGRAM**

EPA 8082 by GC/ECD  
 EMAX Analytical Laboratories, Inc.

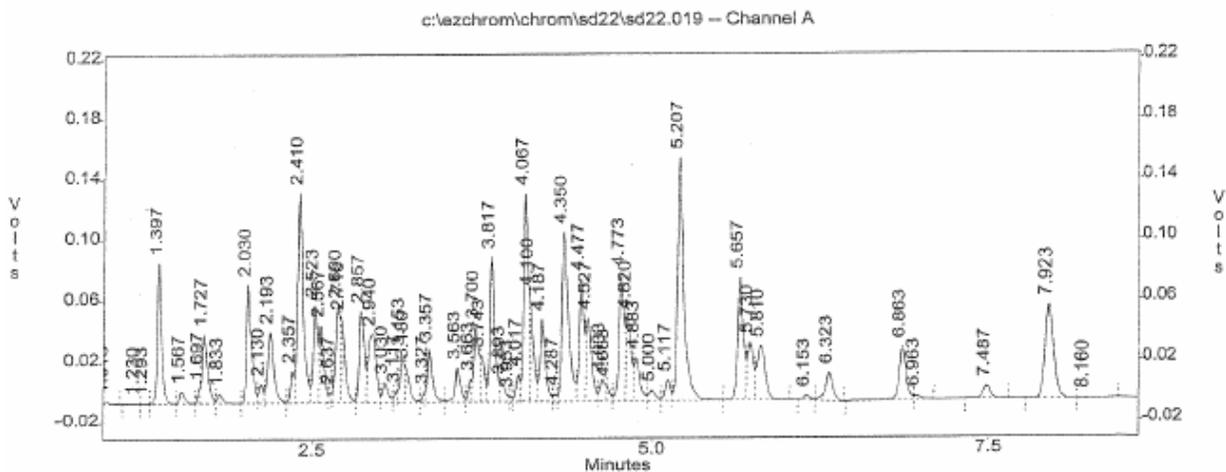
File : c:\ezchrom\chrom\sd22\sd22.019  
 Method : c:\ezchrom\methods\6008d22.met  
 Sample ID : I6008D2201 500 PFB  
 Acquired : Apr 22, 2013 16:28:14  
 Printed : Apr 23, 2013 09:56:16  
 User : Supakit

Channel A Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
10	TCX	1.397	209505	8027.4	26.10	VV
13	1016-1	1.727	117262	1105.5	106.07	SV
15	1016-2	2.030	208123	2115.1	98.40	VV
17	1016-3	2.193	144013	1406.5	102.39	VV
20	1016-4	2.523	147943	1483.4	99.73	VV
25	1016-5	2.857	158991	1530.6	103.88	VV
37	1260-1	3.817	243941	2385.4	102.26	VV
41	1260-2	4.067	324054	3403.5	95.21	VS
45	1260-3	4.350	381390	4078.6	93.51	VV
50	1260-4	4.773	214219	1885.8	113.60	VV
56	1260-5	5.657	244643	2242.9	109.08	VV
64	DCB	7.923	244745	9822.7	24.92	VV

Channel A Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
G1	PCB1016		776332	0.0	510.48	
G2	PCB1260		1408247	0.0	513.66	



**Figure 3A: TYPICAL 1016 / 1260 CHROMATOGRAM**

EPA 8082 by GC/BCD  
 EMAX Analytical Laboratories, Inc.

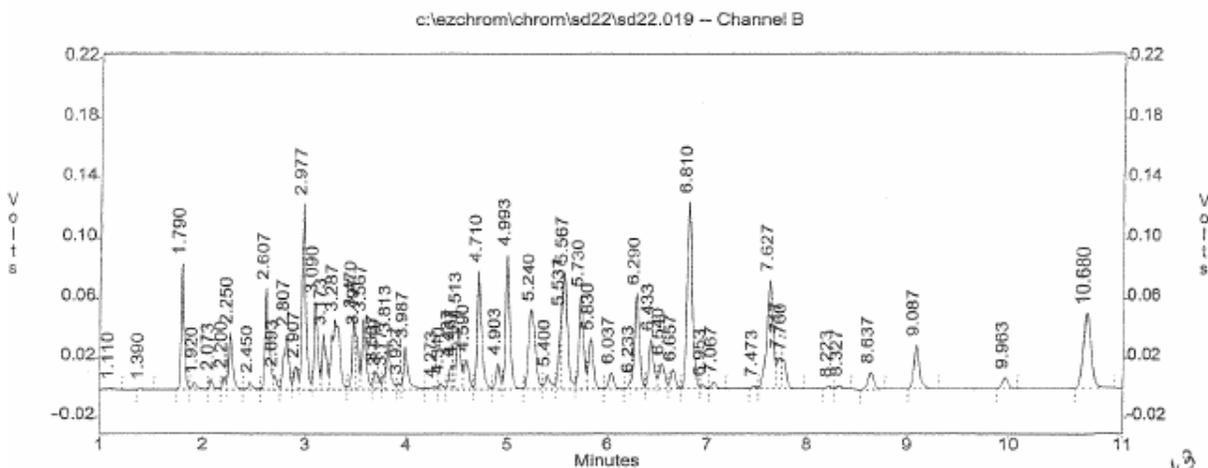
File : c:\ezchrom\chrom\sd22\sd22.019  
 Method : c:\ezchrom\methods\6008d22.met  
 Sample ID : I6008D2201 500 PPB  
 Acquired : Apr 22, 2013 16:28:14  
 Printed : Apr 23, 2013 09:56:16  
 User : Supakit

Channel B Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
7	TCX	1.790	172868	6723.8	25.71	BV
11	1016-1	2.250	96727	909.1	106.40	SV
17	1016-2	2.977	318468	3162.3	100.71	VV
18	1016-3	3.090	146352	1445.4	101.25	VV
21	1016-4	3.470	96205	884.6	108.76	VS
23	1016-5	3.567	138939	1220.9	113.80	VV
36	1260-1	4.710	242972	2525.2	96.22	VV
38	1260-2	4.993	274421	2771.6	99.01	VV
47	1260-3	6.290	219416	1956.9	112.12	SV
51	1260-4	6.810	452413	3935.2	114.97	VV
55	1260-5	7.627	314588	2861.2	109.95	VV
63	DCB	10.680	213337	8570.9	24.89	BB

Channel B Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
G1	PCB1016		796691	0.0	530.92	
G2	PCB1260		1503810	0.0	532.27	



**Figure 3B: TYPICAL 5460 CHROMATOGRAM**

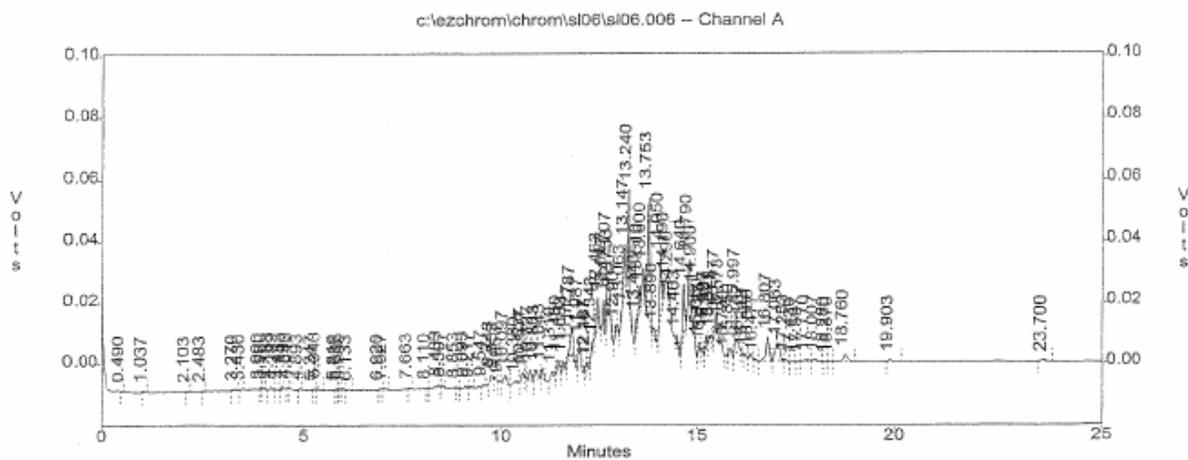
EPA 8082 by GC/ECD  
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\s106\s106.006  
 Method : c:\ezchrom\methods\4608102.met  
 Sample ID : I4608L0202  
 Acquired : Dec 06, 2013 21:01:25  
 Printed : Dec 09, 2013 09:04:41  
 User : Supakit  
 Channel A Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
45	5460-1	11.787	42879	382.9	111.99	vv
57	5460-2	13.240	117349	1162.2	100.98	vv
61	5460-3	13.753	204346	1900.6	107.52	vv
68	5460-4	14.790	76920	737.2	104.34	vv
79	5460-5	15.997	58866	523.6	112.42	vv

Channel A Group Results

#	Peak Name	Ret.Time(min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
G1	PCB5460		500360	0.0	537.24	



**Figure 3B: TYPICAL 5460 CHROMATOGRAM**

EPA 8082 by GC/ECD  
 EMAX Analytical Laboratories, Inc.

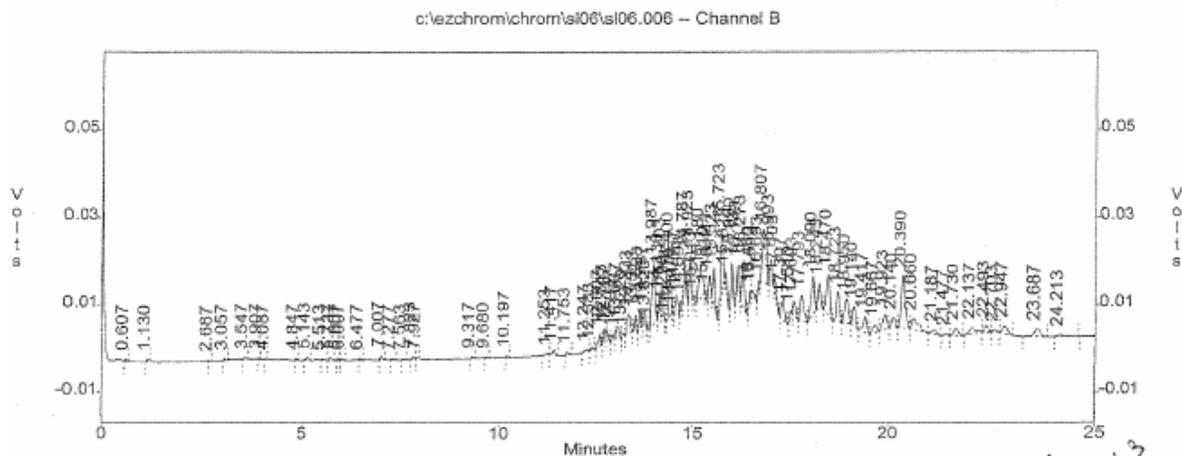
File : c:\ezchrom\chrom\s106\s106.006  
 Method : c:\ezchrom\methods\4608102.met  
 Sample ID : I4608L0202  
 Acquired : Dec 06, 2013 21:01:25  
 Printed : Dec 09, 2013 09:04:42  
 User : Supakit

Channel B Results

#	Peak Name	Ret.Time (min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
43	5460-1	14.400	30916	272.9	113.27	vv
48	5460-2	14.923	34318	314.8	109.03	vv
57	5460-3	16.143	35980	350.9	102.54	vv
71	5460-4	18.470	62839	598.4	105.01	vv
79	5460-5	20.390	88685	788.4	112.49	vv

Channel B Group Results

#	Peak Name	Ret.Time (min)	Area	Ave. CF	ESTD Conc. (ppb)	ICODE
G1	PCB5460		252738	0.0	542.35	



**Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT008 HP-5890  
 GC Column : STX-CLPEST  
 Column size ID : .32MMX30M  
 LFID & Datetime: SD22012A 04/22/13 14:20  
 LFID & Datetime: SD22013A 04/22/13 14:38  
 LFID & Datetime: SD22014A 04/22/13 14:56  
 LFID & Datetime: SD22015A 04/22/13 15:15  
 LFID & Datetime: SD22016A 04/22/13 15:33  
 LFID & Datetime: SD22017A 04/22/13 15:51  
 LFID & Datetime: SD22018A 04/22/13 16:10  
 CONC UNIT: PPB

COMPOUND	CONC	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
	X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X			
PCB1016-1	10.00	1245.80	1264.50	1189.94	1111.22	1010.28	970.48	946.21	1105.489	12.0	
PCB1016-2	10.00	2536.40	2564.95	2311.70	2102.99	1854.57	1753.11	1681.63	2115.051	17.3	
PCB1016-3	10.00	1425.10	1582.55	1504.94	1450.07	1336.32	1287.07	1259.39	1406.492	8.4	
PCB1016-4	10.00	1681.50	1707.10	1566.14	1517.35	1348.08	1288.97	1274.67	1483.401	12.2	
PCB1016-5	10.00	1690.30	1689.80	1631.86	1592.19	1427.36	1351.14	1331.22	1530.552	10.2	
PCB1260-1	10.00	2497.40	2832.70	2665.04	2481.41	2177.64	2050.89	1993.05	2385.447	13.4	
PCB1260-2	10.00	3973.20	3956.45	3780.56	3401.57	3098.52	2868.50	2745.90	3403.528	15.1	
PCB1260-3	10.00	4233.80	4253.40	4335.62	4243.01	3950.23	3785.45	3748.53	4078.577	6.0	
PCB1260-4	10.00	2101.50	2081.20	2016.72	1916.13	1751.73	1651.76	1681.37	1885.773	10.1	
PCB1260-5	10.00	2134.00	2214.35	2293.60	2318.44	2251.94	2258.33	2229.40	2242.865	2.7	
SURROGATE	X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD	
TCK	2.50	7622.80	7776.60	8055.92	8271.16	8180.68	8129.51	8155.13	8027.399	3.0	
DCB	2.50	10453.20	10399.60	10218.48	9930.28	9407.84	9295.45	9054.34	9822.742	5.8	

**Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT008 HP-5890  
 GC Column : STX-CLPESTII  
 Column size ID : .32MMX30M  
 LFD & Datetime: SD22012B 04/22/13 14:20  
 LFD & Datetime: SD22013B 04/22/13 14:38  
 LFD & Datetime: SD22014B 04/22/13 14:56  
 LFD & Datetime: SD22015B 04/22/13 15:15  
 LFD & Datetime: SD22016B 04/22/13 15:33  
 LFD & Datetime: SD22017B 04/22/13 15:51  
 LFD & Datetime: SD22018B 04/22/13 16:10  
 CONC UNIT: PPB

COMPOUND	CONC X	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT								MEAN	%RSD
		1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X			
PCB1016-1	10.00	1034.90	1003.55	953.56	920.08	849.59	802.65	799.27	909.085	10.4	
PCB1016-2	10.00	3364.00	3348.40	3263.74	3246.16	3052.13	2942.87	2918.57	3162.267	5.9	
PCB1016-3	10.00	1584.70	1590.05	1499.06	1500.07	1363.26	1304.63	1275.93	1445.386	9.0	
PCB1016-4	10.00	963.20	981.50	1005.26	968.37	838.92	732.73	702.08	884.580	14.3	
PCB1016-5	10.00	1367.40	1287.35	1139.18	1278.94	1183.28	1143.45	1146.65	1220.892	7.4	
PCB1260-1	10.00	2912.20	2828.30	2682.28	2523.60	2337.13	2210.23	2182.43	2525.167	11.6	
PCB1260-2	10.00	3263.30	3140.35	2941.88	2793.55	2541.98	2376.99	2343.39	2771.633	13.2	
PCB1260-3	10.00	2107.30	2115.55	2022.06	1964.61	1867.61	1817.69	1803.59	1956.917	6.7	
PCB1260-4	10.00	4053.80	4073.20	4062.68	4003.42	3836.75	3766.36	3750.01	3935.174	3.7	
PCB1260-5	10.00	2839.40	2874.40	2904.04	2900.47	2847.30	2830.45	2832.44	2861.214	1.1	
SURROGATE	X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X	MEAN	%RSD	
TCX	2.50	6348.80	6425.00	6637.36	6860.32	6902.80	6900.67	6991.45	6723.771	3.8	
DCB	2.50	8916.00	8977.60	8898.88	8667.16	8279.26	8188.12	8069.01	8570.861	4.5	

**Figure 4: TYPICAL INITIAL CALIBRATION SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : 08 HP-5890  
 GC Column : STX-CLPESTICIDES  
 Column size ID : .32MMID X 30MM X 0.32UM DF  
 LFD & Datetime: SLO2010A 12/02/13 22:05  
 LFD & Datetime: SLO2011A 12/02/13 22:40  
 LFD & Datetime: SLO2012A 12/02/13 23:15  
 LFD & Datetime: SLO2013A 12/02/13 23:50  
 LFD & Datetime: SLO2014A 12/03/13 00:25  
 LFD & Datetime: SLO2015A 12/03/13 00:59  
 LFD & Datetime: SLO2016A 12/03/13 01:34  
 CONC UNIT: PPB

COMPOUND	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT									MEAN	%RSD
	CONC X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X			
PCB5460-1	10.00	345.60	408.85	400.44	387.18	378.08	381.47	378.63	382.893	5.3	
PCB5460-2	10.00	1402.30	1398.15	1239.96	1139.28	1034.54	973.24	947.60	1162.152	16.4	
PCB5460-3	10.00	2122.80	2141.90	1980.30	1872.49	1786.00	1726.26	1674.43	1900.597	9.8	
PCB5460-4	10.00	809.60	825.05	770.08	736.05	694.23	671.68	653.54	737.176	9.1	
PCB5460-5	10.00	499.30	538.00	653.86	496.06	496.83	492.63	488.66	523.620	11.4	

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT008 HP-5890  
 GC Column : STX-CLPESTICIDES II  
 Column size ID : .32MMID X 30MM X 0.25UM DF  
 LFD & Datetime: SLO2010B 12/02/13 22:05  
 LFD & Datetime: SLO2011B 12/02/13 22:40  
 LFD & Datetime: SLO2012B 12/02/13 23:15  
 LFD & Datetime: SLO2013B 12/02/13 23:50  
 LFD & Datetime: SLO2014B 12/03/13 00:25  
 LFD & Datetime: SLO2015B 12/03/13 00:59  
 LFD & Datetime: SLO2016B 12/03/13 01:34  
 CONC UNIT: PPB

COMPOUND	CALIBRATION FACTORS (AREA or HEIGHT)/UNIT									MEAN	%RSD
	CONC X	1.00X	2.00X	5.00X	10.00X	20.00X	30.00X	40.00X			
PCB5460-1	10.00	304.40	303.00	284.96	269.06	256.69	249.62	242.87	272.944	9.2	
PCB5460-2	10.00	350.10	358.55	334.00	310.81	293.42	281.35	275.07	314.758	10.6	
PCB5460-3	10.00	387.10	393.65	364.36	346.38	331.02	319.98	313.67	350.880	9.1	
PCB5460-4	10.00	634.40	657.85	611.10	589.84	573.43	563.00	559.06	598.383	6.3	
PCB5460-5	10.00	826.70	839.15	797.92	775.96	766.59	756.85	755.39	788.364	4.3	

**Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT008 HP-5890  
 GC Column : STX-CLPEST  
 Column size ID : .32MMX30M  
 LFID & Datetime: SD22012A 04/22/13 14:20  
 LFID & Datetime: SD22013A 04/22/13 14:38  
 LFID & Datetime: SD22014A 04/22/13 14:56  
 LFID & Datetime: SD22015A 04/22/13 15:15  
 LFID & Datetime: SD22016A 04/22/13 15:33  
 LFID & Datetime: SD22017A 04/22/13 15:51  
 LFID & Datetime: SD22018A 04/22/13 16:10

COMPOUND	RT OF STANDARDS (MIN)							MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X		FROM	TO	
PCB1016-1	1.727	1.727	1.727	1.727	1.723	1.723	1.723	1.725	1.713	1.737	0.012
PCB1016-2	2.033	2.033	2.033	2.030	2.030	2.030	2.030	2.031	2.020	2.042	0.011
PCB1016-3	2.197	2.193	2.193	2.193	2.190	2.193	2.190	2.193	2.182	2.204	0.011
PCB1016-4	2.523	2.520	2.523	2.520	2.520	2.520	2.520	2.521	2.510	2.532	0.011
PCB1016-5	2.857	2.857	2.857	2.857	2.853	2.857	2.853	2.856	2.845	2.867	0.011
PCB1260-1	3.817	3.813	3.817	3.813	3.813	3.813	3.813	3.814	3.798	3.830	0.016
PCB1260-2	4.070	4.067	4.067	4.067	4.063	4.067	4.063	4.066	4.046	4.086	0.020
PCB1260-3	4.350	4.347	4.350	4.347	4.347	4.347	4.347	4.348	4.327	4.369	0.021
PCB1260-4	4.773	4.773	4.773	4.770	4.770	4.773	4.770	4.772	4.746	4.798	0.026
PCB1260-5	5.657	5.657	5.657	5.653	5.657	5.657	5.653	5.656	5.625	5.687	0.031
SURROGATE	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	RT	FROM	TO	WIDTH
TCX	1.393	1.393	1.397	1.393	1.393	1.393	1.393	1.394	1.383	1.405	0.011
DCB	7.920	7.920	7.923	7.917	7.920	7.920	7.920	7.920	7.874	7.966	0.046

**Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT008 HP-5890  
 GC Column : STX-CLPEST11  
 Column size ID : .32MMX30M  
 LFID & Datetime: SD22012B 04/22/13 14:20  
 LFID & Datetime: SD22013B 04/22/13 14:38  
 LFID & Datetime: SD22014B 04/22/13 14:56  
 LFID & Datetime: SD22015B 04/22/13 15:15  
 LFID & Datetime: SD22016B 04/22/13 15:33  
 LFID & Datetime: SD22017B 04/22/13 15:51  
 LFID & Datetime: SD22018B 04/22/13 16:10

COMPOUND	RT OF STANDARDS (MIN)							MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X		FROM	TO	
PCB1016-1	2.250	2.250	2.250	2.250	2.250	2.250	2.250	2.250	2.239	2.261	0.011
PCB1016-2	2.977	2.977	2.977	2.977	2.973	2.977	2.977	2.976	2.964	2.988	0.012
PCB1016-3	3.090	3.090	3.093	3.090	3.090	3.090	3.090	3.090	3.078	3.102	0.012
PCB1016-4	3.470	3.467	3.470	3.467	3.467	3.467	3.467	3.468	3.452	3.484	0.016
PCB1016-5	3.567	3.563	3.567	3.567	3.563	3.567	3.563	3.565	3.554	3.576	0.011
PCB1260-1	4.713	4.710	4.713	4.710	4.707	4.710	4.707	4.710	4.684	4.736	0.026
PCB1260-2	4.993	4.993	4.993	4.990	4.990	4.993	4.990	4.992	4.968	5.016	0.024
PCB1260-3	6.287	6.287	6.290	6.287	6.287	6.287	6.287	6.287	6.252	6.322	0.035
PCB1260-4	6.810	6.807	6.810	6.807	6.807	6.810	6.810	6.809	6.770	6.848	0.039
PCB1260-5	7.623	7.623	7.627	7.623	7.623	7.627	7.627	7.625	7.579	7.671	0.046
SURROGATE	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X	RT	FROM	TO	WIDTH
TCX	1.787	1.790	1.790	1.790	1.787	1.787	1.787	1.788	1.776	1.800	0.012
DCB	10.677	10.677	10.680	10.677	10.673	10.677	10.680	10.677	10.650	10.704	0.027

**Figure 5: TYPICAL RETENTION TIME WINDOW SUMMARY**

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : 08 HP-5890  
 GC Column : STX-CLPESTICIDES  
 Column size ID : .32MMID X 30MM X 0.32UM DF  
 LFID & Datetime: SLO2010A 12/02/13 22:05  
 LFID & Datetime: SLO2011A 12/02/13 22:40  
 LFID & Datetime: SLO2012A 12/02/13 23:15  
 LFID & Datetime: SLO2013A 12/02/13 23:50  
 LFID & Datetime: SLO2014A 12/03/13 00:25  
 LFID & Datetime: SLO2015A 12/03/13 00:59  
 LFID & Datetime: SLO2016A 12/03/13 01:34

COMPOUND	RT OF STANDARDS (MIN)							MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X		FROM	TO	
PCB5460-1	11.757	11.763	11.767	11.773	11.783	11.787	11.793	11.775	11.763	11.787	0.012
PCB5460-2	13.227	13.233	13.233	13.237	13.240	13.247	13.247	13.238	13.231	13.245	0.007
PCB5460-3	13.737	13.743	13.747	13.750	13.753	13.763	13.763	13.751	13.735	13.767	0.016
PCB5460-4	14.767	14.780	14.780	14.787	14.790	14.803	14.803	14.787	14.775	14.799	0.012
PCB5460-5	15.970	15.983	15.990	15.993	16.003	16.017	16.020	15.997	15.986	16.008	0.011

INITIAL CALIBRATION  
 METHOD EPA 8082

Lab Name : EMAX Inc  
 Instrument ID : GCT00B HP-5890  
 GC Column : STX-CLPESTICIDES II  
 Column size ID : .32MMID X 30MM X 0.25UM DF  
 LFID & Datetime: SLO2010B 12/02/13 22:05  
 LFID & Datetime: SLO2011B 12/02/13 22:40  
 LFID & Datetime: SLO2012B 12/02/13 23:15  
 LFID & Datetime: SLO2013B 12/02/13 23:50  
 LFID & Datetime: SLO2014B 12/03/13 00:25  
 LFID & Datetime: SLO2015B 12/03/13 00:59  
 LFID & Datetime: SLO2016B 12/03/13 01:34

COMPOUND	RT OF STANDARDS (MIN)							MEAN RT	RT WINDOW		RTWINDOW WIDTH
	1.0X	2.0X	5.0X	10.0X	20.0X	30.0X	40.0X		FROM	TO	
PCB5460-1	14.380	14.390	14.393	14.393	14.400	14.413	14.413	14.397	14.388	14.406	0.009
PCB5460-2	14.900	14.913	14.917	14.920	14.927	14.940	14.940	14.922	14.911	14.933	0.011
PCB5460-3	16.120	16.133	16.140	16.143	16.153	16.167	16.170	16.147	16.132	16.162	0.015
PCB5460-4	18.443	18.460	18.470	18.470	18.497	18.507	18.513	18.480	18.460	18.500	0.020
PCB5460-5	20.363	20.373	20.393	20.397	20.423	20.433	20.440	20.403	20.366	20.440	0.037

**Figure 6: TYPICAL SAMPLE RESULT SUMMARY**

METHOD 3546/8082  
 PCBs

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=====
Client       : XYZ, INC.
Project      : CLEAN LAND
Batch No.    : 14E102
Sample ID    : BVBS1265S001
Lab Samp ID  : E102-03
Lab File ID  : SE22008A
Ext Btch ID : CPE022S
Calib. Ref. : SE22002A

Date Collected: 05/13/14
Date Received: 05/14/14
Date Extracted: 05/20/14 13:18
Date Analyzed: 05/22/14 19:37
Dilution Factor: 1
Matrix       : SOIL
% Moisture   : 3.6
Instrument ID : GCT008
=====
  
```

PARAMETERS	RESULTS (ug/kg)	RL (ug/kg)	MDL (ug/kg)	
AROCLOR 1016	(ND)   ND	18	8.8   8.8	
AROCLOR 1221	(ND)   ND	34	18   18	
AROCLOR 1232	(ND)   ND	18	8.8   8.8	
AROCLOR 1242	(ND)   ND	18	8.8   8.8	
AROCLOR 1248	(ND)   ND	18	8.8   8.8	
AROCLOR 1254	(ND)   ND	18	8.8   8.8	
AROCLOR 1260	(ND)   ND	18	8.8   8.8	
AROCLOR 1262	(ND)   ND	34	18   18	
AROCLOR 1268	(ND)   ND	34	18   18	
AROCLOR 5432	(ND)   ND	52	26   26	
AROCLOR 5442	(ND)   ND	52	26   26	
AROCLOR 5460	(ND)   ND	52	26   26	
SURROGATE PARAMETERS				
DECACHLOROBIPHENYL	(14.21)   12.68	SPK_AMT 13.83	% RECOVERY (103)   91.7	QC LIMIT 45-120

Left of | is related to first column ; Right of | related to second column  
 Final result indicated by ( )  
 \* out side of QC Limit

**Figure 7: TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN LAND  
 BATCH NO.: 14E102  
 METHOD: METHOD 3546/8082

MATRIX: SOIL % MOISTURE: NA  
 DILUTION FACTOR: 1 1  
 SAMPLE ID: MBLK1S  
 LAB SAMP ID: 60E0225B 60E0225L  
 LAB FILE ID: SE22005A SE22006A  
 DATE EXTRACTED: 05/20/1413:18 05/20/1413:18 DATE COLLECTED: NA  
 DATE ANALYZED: 05/22/1417:52 05/22/1418:27 DATE RECEIVED: 05/20/13  
 PREP. BATCH: CPE0225 CPE0225  
 CALIB. REF: SE22002A SE22002A

ACCESSION:

PARAMETER	BLNK RSLT (ug/kg)	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BS % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Aroclor 1016	(ND) ND	167	(171) 163	(103) 98	167	(173) 161	(104) 96	(1) 1	50-150	30
Aroclor 1260	(ND) ND	167	159 (162)	95 (97)	167	158 (165)	95 (99)	1 (2)	50-150	30
Aroclor 5460	(ND) ND	83.3	(88.3) 84.9	(106) 102	83.3	(90.0) 86.1	(108) 103	(2) 1	50-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	QC LIMIT (%)
Decachlorobiphenyl	13.33	16.87 (14.87)	127* (112)	13.33	16.13 15.01	121* (113)	45-120

**Figure 8: TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA MS/MSD ANALYSIS										
CLIENT:	XYZ, INC.									
PROJECT:	CLEAN LAND									
BATCH NO.:	14E102									
METHOD:	METHOD 3546/8082									
=====										
MATRIX:	SOIL			% MOISTURE:		3.6				
DILUTION FACTOR:	1	1	1							
SAMPLE ID:	BVBS1265S001									
LAB SAMP ID:	E102-03	E102-03M	E102-03S							
LAB FILE ID:	SE22008A	SE22009A	SE22010A							
DATE EXTRACTED:	05/20/1413:18	05/20/1413:18	05/20/1413:18	DATE COLLECTED:	05/13/14					
DATE ANALYZED:	05/22/1419:37	05/22/1420:11	05/22/1420:46	DATE RECEIVED:	05/14/14					
PREP. BATCH:	CPE022S	CPE022S	CPE022S							
CALIB. REF:	SE22002A	SE22002A	SE22002A							
ACCESSION:										
PARAMETER	SMPL RSLT (ug/kg)	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Aroclor 1016	(ND) ND	173	(155) 176	(90) 102	173	(163) 152	(94) 88	(4) 15	29-135	30
Aroclor 1260	(ND) ND	173	(165) 165	(95) 95	173	(164) 141	(95) 82	(1) 16	29-135	30
Aroclor 5460	(ND) ND	86.4	90.1 (93.3)	104 (108)	86.4	(83.9) 79.5	(97) 92	(7) 16	29-135	30
=====										
SURROGATE PARAMETER	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	QC LIMIT (%)			
Decachlorobiphenyl	13.83	(16.36) 14.64	(118) 106	13.83	(14.44) 12.73	(104) 92.1	45-120			

**Figure 9: TYPICAL CASE NARRATIVE**

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CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN LAND

SDG : 14E102

METHOD 3520C/3546/8082  
PCBs

A total of six (6) soil samples were received on 05/14/14 for PCBs and PCTs analysis, Method 3546/8082 in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blanks were analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample

A set of LCS/LCD and lab control sample was analyzed with the samples in this SDG.

Percent recoveries for 60E022SL/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the samples in this SDG.

Percent recoveries for E102-03M/S were within project QC limits.

Surrogate

Surrogate was added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Sample extracts subjected to appropriate cleanup technique to reduce matrix interference are recorded in extraction log.

**Table 1: INTERMEDIATE STANDARD PREPARATION**

Standard Name	Stock Standard Conc. (ppm)	Preparation			Final Conc. (µg/L)
		Aliquot (ml)	Solvent	Final Vol. (ml)	
PCB 1016	1000	0.4	Hexane	100	4000
PCB 1260	1000	0.4	Hexane	100	4000
PCB 1221	1000	0.4	Hexane	100	4000
PCB 1232	1000	0.4	Hexane	100	4000
PCB 1242	1000	0.4	Hexane	100	4000
PCB 1248	1000	0.4	Hexane	100	4000
PCB 1254	1000	0.4	Hexane	100	4000
PCB 1262	1000	0.4	Hexane	100	4000
PCB 1268	1000	0.4	Hexane	100	4000
PCT 5460	1000	0.4	Hexane	100	4000
PCT 5432	1000	0.4	Hexane	100	4000
PCT5442	1000	0.4	Hexane	100	4000
<b>Surrogate</b>					
TCX	200	0.1	Hexane	100	200
DCB	200	0.1	Hexane	100	200

**Table 2: INITIAL CALIBRATION STANDARD PREPARATION**

Standard #	Standard Name	Intermediate Std. (µg/L)	Preparation			Final Conc. (µg/L)
			Aliquot (µl)	Solvent	Final Vol. (µl)	
1	PCB 1660 / PCT 5460	4000	10	Hexane	800	50
	Surrogate	200				2.5
2	PCB 1660 / PCT 5460	4000	20	Hexane	800	100
	Surrogate	200				5
3	PCB 1660 / PCT 5460	4000	50	Hexane	800	250
	Surrogate	200				12.5
4	PCB 1660 / PCT 5460	4000	100	Hexane	800	500
	Surrogate	200				25
5	PCB 1660 / PCT 5460	4000	200	Hexane	800	1000
	Surrogate	200				50
6	PCB 1660 / PCT 5460	4000	300	Hexane	800	1500
	Surrogate	200				75
7	PCB 1660 / PCT 5460	4000	400	Hexane	800	2000
	Surrogate	200				100

**Table 3: CALIBRATION CHECK STANDARD PREPARATION**

Standard Name	Intermediate Standard Conc. (µg/L)	Preparation			Final Conc. (µg/L)
		Aliquot (µl)	Solvent	Final Vol. (µl)	
PCB 1016	4000	100	Hexane	800	500
PCB 1260	4000	100	Hexane	800	500
PCB 1221	4000	100	Hexane	800	500
PCB 1232	4000	100	Hexane	800	500
PCB 1242	4000	100	Hexane	800	500
PCB 1248	4000	100	Hexane	800	500
PCB 1254	4000	100	Hexane	800	500
PCB 1262	4000	100	Hexane	800	500
PCB 1268	4000	100	Hexane	800	500
PCT 5460	4000	100	Hexane	800	500
PCT 5432	4000	100	Hexane	800	500
PCT 5442	4000	100	Hexane	800	500
<b>Surrogate</b>					
TCX	200	100	Hexane	800	25
DCB	200	100	Hexane	800	25

**Table 4: SPIKE STANDARD AND SURROGATE STANDARD PREPARATION**

Standard Name	Stock Standard Conc. (mg/L)	Preparation			Final Conc. (µg/L)
		Aliquot (ml)	Solvent	Final Vol. (ml)	
PCB 1016	1000	1.0	50% Isopropanol 50% Hexane	100	10,000
PCB 1260	1000	1.0	50% Isopropanol 50% Hexane		
PCT 5460	1000	1.0	50% Isopropanol 50% Hexane	100	10,000
<b>Surrogate</b>					
TCX	200	2	50% Isopropanol 50% Hexane	1000	400
DCB	200	2	50% Isopropanol 50% Hexane	1000	400

**Table 5: COMPOUND LIST**

Analytes		Surrogates
PCB 1016	PCT 5432	Decachlorobiphenyl
PCB 1221	PCT 5442	Tetrachloro-m-xylene
PCB 1232	PCT 5460	
PCB 1242		
PCB 1248		
PCB 1254		
PCB 1260		
PCB 1262		
PCB 1268		

**Table 6: ESTABLISHED LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTITATION (LOQ)**

ANALYTES	AQUEOUS (µg/L)			SOIL (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
PCB 1016	0.45	0.45	0.45	13.2	16.7	33.3
PCB 1221	0.29	0.29	0.29	8.3	16.7	33.3
PCB 1232	0.25	0.25	0.25	9.0	16.7	33.3
PCB 1242	0.25	0.25	0.25	9.3	16.7	33.3
PCB 1248	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1254	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1260	0.31	0.31	0.31	9.9	16.7	33.3
PCB 1262	0.25	0.25	0.25	8.3	16.7	33.3
PCB 1268	0.25	0.25	0.25	8.3	16.7	33.3
PCT 5432	0.5	0.5	0.5	10	20	40
PCT 5442	0.5	0.5	0.5	10	20	40
PCT 5460	0.5	0.5	0.5	10	20	40
Tetrachloro-m-xylene	0.020	0.020	0.020	0.42	0.83	1.67
Decachlorobiphenyl	0.025	0.025	0.025	0.42	0.83	1.67

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURES**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Min. 5 Point Initial Calibration for PCB 1016 + 1260 and PCT 5460	Initially, as needed	RSD for all analytes ≤ 20%. Linear – least squares regression $r \geq 0.995$ .	Correct then problem then repeat initial calibration.		
Second-source Calibration Verification for PCB 1016/1260 and PCT 5460	Once per 5-point initial calibration	All analytes within ± 20% of expected value from the ICAL.	Correct the problem. If problem persists, repeat initial calibration.		
Initial Calibration Verification Check for PCB 1016/1260 and PCT 5460	Daily, before sample analysis	All analytes within ± 20%	Correct the problem. If problem persists, repeat initial calibration		
Calibration verification for PCB 1016/1260 and PCT 5460	After every 12 hours and at the end of the analytical sequence	All analytes within ± 20% of expected value	Correct the problem then repeat initial calibration verification and re-analyze all samples since last successful calibration verification.		
Method Blank	One per preparation batch (≤ 20 samples per matrix)	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Re-prep and re-analyze method blank and all samples processed with the contaminated blank		
LCS (PCB 1016/1260 and PCT 5460)	One LCS per analytical batch (≤20 samples per matrix)	Within project QC Limits	Correct the problem, then re-prep and re-analyze the LCS and all associated samples.		
Surrogate Spike	Every sample, spiked sample, standard, and method blank	Within project QC Limits	Correct the problem then re-extract and re-analyze sample		
Matrix Spike/ Matrix Spike Duplicate (PCB 1016/1260 and PCT 5460)	One MS/MSD per 20 project samples per matrix	Within project QC Limits	If chromatogram is indicative of matrix interference, discuss in case narrative. Otherwise, check for probable source of error and perform corrective action as necessary.		
Confirmation	100% for all positive results	Same as primary column	If quantitation criteria are not met, use confirmation for qualitative identification only.		
Comments: 1. For flagging criteria refer to PSR. Otherwise, if MB is non-compliant, apply “B” to specific analyte(s) on all associated samples, apply “J” to all values between LOD and LOQ.				Reviewed By:	
				Date:	

**Appendix 2:**

**DEMONSTRATION OF CAPABILITY**

DEMONSTRATION OF CAPABILITY  
PCBs  
METHOD: EPA 8082/ 8082A

**MATRIX: WATER**

Analytical SOP: EMAX-8082 Rev. 4  
Sample Preparation SOP: EMAX-3520 Rev. 5  
Conc Unit: µg/L  
Sample Amount(mL): 1000  
Extract Volume (mL): 10

Instrument ID: 08  
Extraction batches: CPE002W - 5/5/14  
CPE009W - 5/7/14  
Extracted by: J. Muertigue  
Analysis date: 5/7/14 & 5/14/14  
Analyzed by: R. Zhou

PARAMETER	60E002WL	60E002WC	60E009WL	60E009WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SE07006A	SE07007A	SE12012A	SE12013A							
PCB 1016	5.11	4.91	5.44	4.66	5	5.03	101	0.3	7	60 - 130	PASSED
PCB 1260	4.65	4.44	5.03	4.46	5	4.65	93	0.3	6	70 - 130	PASSED
PCT 5460	2.34	2.45	2.42	2.41	2.5	2.41	96	0.05	2	50 - 150	PASSED
TCX	0.396	0.385	0.399	0.391	0.4	0.393	98	0.006	2	60 - 130	PASSED
DCB	0.406	0.402	0.433	0.432	0.4	0.418	105	0.016	4	70 - 130	PASSED

PARAMETER	60E002WL	60E002WC	60E009WL	60E009WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SE07006B	SE07007B	SE12012B	SE12013B							
PCB 1016	5.02	4.83	4.86	4.69	5	4.85	97	0.1	3	60 - 130	PASSED
PCB 1260	4.70	4.68	4.86	4.76	5	4.75	95	0.1	2	70 - 130	PASSED
PCT 5460	2.17	2.28	2.16	2.14	2.5	2.19	88	0.1	3	50 - 150	PASSED
TCX	0.409	0.399	0.405	0.407	0.4	0.405	101	0.004	1	60 - 130	PASSED
DCB	0.372	0.367	0.389	0.384	0.4	0.378	95	0.010	3	70 - 130	PASSED

**MATRIX: SOIL**

Analytical SOP: EMAX-8082 Rev. 4  
Sample Preparation SOP: EMAX-3550 Rev. 4  
Conc Unit: µg/Kg  
Sample Amount(g): 30  
Extract Volume (mL): 10

Instrument ID: 08  
Extraction batches: CPF0065 - 6/5/14  
CPF0185 - 6/10/14  
Extracted by: J. Villena  
Analysis date: 6/6/14 & 6/10/14  
Analyzed by: R. Zhou

PARAMETER	60F0065L	60F0065C	60F0185L	60F0185C	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SF06006A	SF06007A	SF10006A	SF10007A							
PCB 1016	181	171	163	163	167	169.56	102	8.5	5	70 - 130	PASSED
PCB 1260	159	154	156	155	167	155.63	93	2.2	1	70 - 140	PASSED
PCT 5460	76.5	77.4	84.3	82.9	83.3	80.29	96	3.9	5	50 - 150	PASSED
TCX	14.6	13.6	14.3	14.4	13.3	14.251	107	0.421	3	50 - 140	PASSED
DCB	14.2	14.1	14.9	14.8	13.3	14.508	109	0.446	3	70 - 140	PASSED

PARAMETER	60F0065L	60F0065C	60F0185L	60F0185C	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SF06006B	SF06007B	SF10006B	SF10007B							
PCB 1016	190	184	164	170	167	176.93	106	12.0	7	70 - 130	PASSED
PCB 1260	161	159	168	168	167	164.13	98	4.7	3	70 - 140	PASSED
PCT 5460	71.8	73.2	77.5	76.8	83.3	74.84	90	2.8	4	50 - 150	PASSED
TCX	16.2	15.5	15.7	15.7	13.3	15.756	118	0.305	2	50 - 140	PASSED
DCB	12.2	12.1	13.4	13.2	13.3	12.733	95	0.657	5	70 - 140	PASSED







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SOP REVIEW FORM

EMAX-8260  
SOP No.

Rev. 10  
Revision Number

VOLATILE ORGANICS BY GC/MS  
Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

No revision needed.

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

  
FARINA MADAMBA

Date:

6/4/15

## STANDARD OPERATING PROCEDURES

VOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-14

Prepared By: Souzan Greas *Souzan Greas* Date: 06-04-14

Approved By: Kenette Pimentel *KP* Date: 06.04.14  
QA Manager

Approved By: Caspar Pang *C Pang* Date: 06-04-14  
Laboratory Director

**Control Number: 8260-10-**

**1.0 SCOPE AND APPLICATION**

- 1.1. This analytical method is used to determine the concentration of volatile organic compounds whose boiling points are below 200°C and are water insoluble or slightly water-soluble found in solid or liquid samples. The list of compounds is summarized in Tables 6 and 7. Additional analytes may be added after verification.
- 1.2. This SOP is an adaptation of SW846 Method 8260B.

**2.0 SUMMARY OF METHOD**

- 2.1. A measured sample is extracted using a purge and trap concentrator system. The extract is introduced to a temperature-programmed GC. The analytes are eluted through the GC column separating each analyte relative to its volatility. These analytes are captured and ionized by the mass spectrometer. The ionized fragments are measured by mass to charge ratio. Analyte qualitative identification is based on the characteristic electron impact mass spectra. Analyte quantitative identification is based on the response of the major ion relative to an internal standard using a multi-point calibration curve.
- 2.2. **Interferences**
  - 2.2.1. Contamination may occur by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through sample container septum during shipment and storage. Trip blanks and storage blanks can serve as means of monitoring.
  - 2.2.2. Glassware and other sample processing materials in which the samples come into contact with are possible sources of contamination. All glassware and other materials used must be purchased pre-cleaned or decontaminated prior to use.
  - 2.2.3. Solvents and reagents are possible sources of contamination. All solvents and reagents must be GC grade and must pass the QC checks prior to use.
  - 2.2.4. Contamination by carry-over can occur whenever high concentration samples are analyzed in sequence with a low concentration sample. To reduce potential carry-over, the concentrator must be thoroughly baked-out between samples and the sample syringe and purging device must be thoroughly rinsed with an appropriate solvent between samples.
  - 2.2.5. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

**3.0 DETECTION LIMITS**

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-14

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- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Refer to Table 6 and Table 7 for established DL, LOD and LOQ levels.

**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and requires dilution to properly quantitate.
- 4.2. The concentration in the diluted sample should be at or above the project reporting limit. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.
- 4.3. **Typical Dynamic Range**
  - 4.3.1. Water: 5 µg/L to 200 µg/L (5 ml purge)  
1 µg/L to 40 µg/L (25 ml purge)
  - 4.3.2. Soil: 5 µg/kg to 200 µg/kg

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1. Aqueous Samples**

- 5.1.1. Samples received in the laboratory are expected to be contained in 40 ml vials with teflon lined septa with zero headspace.  
  
*Note: The size of any bubble caused by degassing upon cooling the sample must not exceed 6 mm.<sup>1</sup>*
- 5.1.2. Samples must be stored at ≤ 6°C without freezing.
- 5.1.3. Samples preserved in HCL must be analyzed within 14 days from the date of sampling. Samples with no chemical preservative must be analyzed within 7 days from the date of sampling.
- 5.1.4. If Acrolein and Acrylonitrile are target analytes, samples must be analyzed within 14 days if preserved with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> to pH 4-5. Samples received unpreserved must be analyzed within 3 days from sampling date<sup>2</sup>.

**5.2. Soil Samples**

- 5.2.1. Samples received in glass jars or brass tubes must be stored at ≤ 6°C without freezing. Samples for low level and extracted in methanol for high level must be analyzed within 14 days from sampling date.
- 5.2.2. Samples received in encore tubes may be frozen, preserved with sodium bisulfate or extracted with methanol prior to analysis.

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<sup>1</sup> Referenced from SW846 Method 5030B, Section 6.1.

<sup>2</sup> Reference: 40CFR Table 11 Footnote 10

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-14

- Encore samples to be frozen must be analyzed within 14 days from sampling date.
- Encore samples to be preserved with sodium bisulfate for low level and extracted with methanol for high level must be done within 48 hours and analyzed within 14 days from sampling date.
- Preserved samples and extracts must be stored at  $\leq 6^{\circ}\text{C}$  without freezing.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-5030 Purge and Trap
- 6.2. EMAX-5035 Closed-System Purge and Trap
- 6.3. EMAX-DM01 Data Flow and Review
- 6.4. EMAX-QA04 Detection Limit (DL)
- 6.5. EMAX-QA05 Training
- 6.6. EMAX-QA08 Corrective Action
- 6.7. EMAX-QC01 Quality Control for Chemicals
- 6.8. EMAX-QC02 Analytical Standard Preparation
- 6.9. EMAX-QC07 Glassware Cleaning
- 6.10. EMAX-SM01 Sample Management
- 6.11. EMAX-SM03 Waste Disposal
- 6.12. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all SDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of the body, rinse the affected body part thoroughly with copious amounts of water. If irritations or any other discomfort related to the incident persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS & REAGENTS****8.1. Instruments and Supplies**

Gas Chromatography	HP 5890 Series II or equivalent
Detector	HP 5971 MSD or equivalent

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-14

Column	RTX 502.2 (0.32 mm x 60 m), 1.8 µm thickness or equivalent after verification that the four gases (chloromethane, bromomethane, chloroethane, and vinyl chloride) can be resolved > 90% from each other in the total ion chromatogram
Data Acquisition Software	ChemStation or equivalent
Purge & Trap Device	OI 4560/Encon Evolution/EST or equivalent
Multiple purging module	Archon/Centurion or equivalent
Gases	Ultra-high purity helium/Air
Syringes	5 ml, 25 ml Luerlok gas-tight
Microsyringes	1, 10, 20, 25, 50, 100 and 1000 µl (Hamilton 702N or equivalent)
Volumetric Flasks	2, 5, 10, 50 and 100 ml with ground glass stopper
Heated Sparge	Archon or Automatic sample heating jacket or equivalent

8.2. **Chemicals and Reagents**

Extraction Solvent	Purge & Trap Grade Methanol or equivalent
Reagent Water	Organic-free water
Reagent Soil	Organic-free Ottawa Sand or equivalent
Preservative	Sodium Bisulfate

9.0 **STANDARDS**

9.1. Standard preparation for VOA is summarized in Tables 1 to 4. Refer to EMAX-QC02 for proper analytical standard preparation and EMAX-SM04 for proper labeling. Other concentration levels may be prepared as long as it complies with the method and/or project requirements.

9.2. **Stock Standard**

- 9.2.1. Purchase Stock Standards as certified solutions.
- 9.2.2. Purchase one set of calibration standard (refer to Table 1) for calibration and a secondary source Stock Standard for calibration verification (refer to Table 2).
- 9.2.3. Purchase Surrogate Mix at 2500 mg/L and Internal Standard at 2500 mg/L (refer to Table 3).
- 9.2.4. Purchase BromoFluorobenzene (BFB) as Tuning Standard at 5000 mg/L (refer to Table 4).
- 9.2.5. After opening, transfer in inert vials with minimal headspace and store at -10°C to -20°C.

9.3. **Intermediate Standards**

- 9.3.1. Using the stock standard solutions, prepare intermediate standards in methanol according to Tables 1 to 4 and store with minimal headspace in an inert vial.

9.4. **Initial Calibration Standards (ICAL)**

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-149.4.1. ICAL for 5 ml Purge

9.4.1.1. Using intermediate standards (refer to Tables 1 and 3), prepare multi calibration standards (minimum of five different concentrations) in reagent water as suggested below.

Calibration Pt.	VOA (µg/L)*	Surrogate (µg/L)	Internal Std (µg/L)
1	5	5	50
2	10	10	50
3	20	20	50
4	50	50	50
5	100	100	50
6	200	200	50

\* *Ketones, Acrolein, Acrylonitrile and tert-Butanol are 5X the indicated concentration and m/p-Xylene is 2x the indicated concentration.*

9.4.2. ICAL for 25 ml Purge

9.4.2.1. Using intermediate standards (refer to Tables 1 and 3), prepare multi calibration standards (minimum of five different concentrations) in reagent water as suggested below:

Calibration Pt.	VOA (µg/L)*	Surrogate (µg/L)	Internal Std (µg/L)
1	0.5	0.5	10
2	1	1	10
3	2	2	10
4	10	10	10
5	20	20	10
6	40	40	10

\* *Ketones, Acrolein, Acrylonitrile and tert-Butanol are 5X the indicated concentration and m/p-Xylene is 2x the indicated concentration.*

9.5. **Initial Calibration Verification Standard (ICV)**

9.5.1. Using the Intermediate Standard prepared from the secondary source (refer to Tables 2 and 3), spike into 5 ml or 25 ml purge in reagent water as suggested below.

9.5.1.1. ICV for 5 ml purge

ICV	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

\* *Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-149.5.1.2. ICV for 25 ml purge

ICV	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25 ml	10	10	10

*\* Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.6. **Daily Calibration Check Standard (DCC)**

9.6.1. Using the Intermediate Standard prepared from the same source as the ICAL Standard (refer to Tables 1 and 3), spike into 5 ml or 25 ml purge in reagent water as suggested below.

9.6.1.1. DCC for 5 ml purge

DCC	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

*\* Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.6.1.2. DCC for 25 ml purge

DCC	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25	10	10	10

*\* Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

9.7. **LCS and Matrix Spike Standard**

9.7.1. For spike standards, use the Intermediate Standard prepared from the secondary source (refer to Tables 2 and 3), spike into the 5 ml or 25 ml purge sample as suggested below (unless otherwise specified by the project). Spike 5 ml or 25 ml reagent water for LCS water or 5 g reagent soil in 5 ml reagent water for LCS soil.

9.7.1.1. LCS and Matrix Spike for 5 ml purge

LCS or MS/MSD	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
5 ml	50	50	50

*\* Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

## STANDARD OPERATING PROCEDURES

**VOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8260 Revision No. 10 Effective Date: 06-Jun-149.7.1.2. LCS and Matrix Spike for 25 ml purge

LCS or MS/MSD	VOA* (µg/L)	Surrogate (µg/L)	Internal Standard (µg/L)
25 ml	10	10	10

*\* Ketones, Acrolein, Acrylonitrile and Tert-Butanol are 5x the indicated concentration and M/P-xylene is 2X the indicated concentration.*

**10.0 PROCEDURES****10.1. Sample Preparation**

10.1.1. For aqueous samples, refer to EMAX-5030.

10.1.1.1. Check the pH and presence of residual chlorine from remaining sample. Record samples with pH  $\geq 2$  and residual chlorine  $\geq 5$  mg/L in the analysis log.

10.1.2. For soil samples, refer to EMAX-5035.

**10.2. Instrument Parameters**

10.2.1. From the main gas supply (gas Tanks) regulate gas pressure at 80 psi.

10.1.1. Fine-tune the instrument guided by the parameter conditions suggested below. Adjust the parameter conditions accordingly to obtain optimum condition. Print the instrument parameter and post it on the instrument for daily routine maintenance check.

10.2.2. Typical GC Parameters

Carrier gas flow (column) helium	1 – 5 ml/min
Initial Temp	35°C; hold for 1 min.
Rate 1	8°C/min. to 160°C/min
Rate 2	30°C/min to 230°C/min; hold for 3 min.
Inject Port	200°C
Interface	250°C

10.2.3. Mass Spectrometer Parameter

Scan Start	0.5 min.
Mass Range	35 to 300
Multiplier	1200 to 2700

10.2.4. Typical Purge and Trap Condition

10.2.4.1. Purge samples at 40°C for 11 minutes, desorbed at 250°C for 2 minutes and then bake the trap at 260°C for 11 minutes.

**10.3. Calibration**

10.3.1. Set GC/MS operating condition as described in Section 10.2.

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10.3.2. Perform Tune Check

- 10.3.2.1. Introduce a BFB to yield 5 – 50 ng by either direct injection or purge and trap in 5 ml or 25 ml organic-free water (using tuning standard). Refer to table 4.
- 10.3.2.2. Evaluate the tune check by the highest scan on the peak or the average of at least 3 scans (before, at and after the apex) with a background subtraction using a single scan no more than 20 scans prior to the elution of BFB.
- 10.3.2.3. Check Table 5 for acceptance criteria or follow the manufacturer's recommendation for tuning. A valid tune check expires after 12 hours.
- 10.3.2.4. If non-compliant refer to Section 12 for corrective action.

10.3.3. Initial Calibration (ICAL)

- 10.3.3.1. Perform ICAL when one of the conditions occurs.
  - Instrument is new
  - Instrument undergoes a major repair
  - DCC failed to meet the acceptance criteria
- 10.3.3.2. Optimize the instrument condition prior to ICAL
  - Ensure that instrument parameters are set up properly
  - Ensure that there is no evidence of leak
  - Ensure that instrument maintenance is performed on schedule
  - Ensure that instrument tune check and column performance is not indicative that it is at the threshold of failing the acceptance criteria
- 10.3.3.3. Analyze a multi-point initial calibration curve as suggested in Figure 3 after a valid tune check.
- 10.3.3.4. Base quantitation of identified compounds on the integrated abundance from the EICP of the assigned primary characteristic ion (refer to Tables 6 and 7). For optimum output, assign internal standard to each compound based on the nearest retention time or as suggested on Tables (6 and 7).
- 10.3.3.5. Evaluate the ICAL Acceptance
  - 10.3.3.5.1. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:
    - Check the established retention time window
    - Check the relative intensity of major ions
    - Adjust accordingly if necessary
  - 10.3.3.5.2. Evaluate retention time of each analyte with respect to the nearest internal standard. The relative retention time (RRT) of each analyte should agree within  $\pm 0.06$  RRT units.

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10.3.3.5.3. At a minimum, evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.3.5.4. Check RSD and correlation coefficient. If more than 10% of the compounds included with the initial calibration exceed the 15% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform necessary instrument maintenance and repeat calibration. Refer to 10.3.3.2, Section 12 for corrective action.

10.3.3.6. Application of ICAL Curve for Quantitation

10.3.3.6.1. Generate a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRFm), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq.-10.5.1.1, Eq.-10.5.1.2, Eq.-10.5.1.6 and Eq.-10.5.1.7 respectively.

10.3.3.6.2. If RSD is  $\leq 15\%$  average response factor may be applied.

10.3.3.6.3. Apply Inverse Weighting Factor ( $1/y$  or  $1/y^2$ ;  $y$  being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 15\%$  and correlation coefficient of  $\geq 0.995$ .

10.3.3.6.4. Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 15\%$  and correlation coefficient of  $\geq 0.995$ .

10.3.3.6.5. It may be appropriate to force the regression through zero for specific analytes<sup>3</sup>. When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Analyze ICV to verify the concentration of the ICAL standards (refer to Section 9.5).

10.3.4.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of  $\pm 30$  seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two ( $-50\%$  to  $+100\%$ ).

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<sup>3</sup> SW846 Method 8000B, Section 7.5.3

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10.3.4.5. Refer to Appendix 1 for ICV acceptance criteria and/or corrective action.

10.3.4.6. When non-compliant refer to Section 12 for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC to check the validity of the ICAL (refer to 9.6).

10.3.5.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.5.3. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.5.4. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of  $\pm 30$  seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.5.5. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.6. Establish RRF of each analyte, calculate %D (Eq.-10.5.2.1) against the ICAL.

10.3.5.7. Refer to Appendix 1 for DCC acceptance criteria and/or corrective action.

10.3.5.8. When non-compliant refer to Section 12 for corrective action.

**10.4. Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Analyze BFB and evaluate tuning

10.4.1.2. Analyze DCC and check ICAL validity

10.4.1.3. Analyze Lab Control Sample

10.4.1.4. Analyze Lab Control Sample Duplicate (if required)

10.4.1.5. Analyze Method Blank

10.4.1.6. Analyze samples to a maximum number of 12-hours from the time of BFB injection.

10.4.1.7. Analyze a pair of matrix spikes (MS/MSD) for every 20 samples of the same matrix.

10.4.1.8. Record analytical sequence in the analysis log.

10.4.2. Sample Result Evaluation

10.4.2.1. Check the QC criteria as soon as the data is available.

✓ Check method blank. If result is non-compliant and analyte in question is not detected in any sample or contamination is  $< 10X$  of the sample concentration, results maybe reportable. Verify with the PM if results can be reported.

✓ Compare the retention times of each Internal Standards (IS) to the ICAL mid point (must be  $\pm 30$  seconds).

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- ✓ Compare the area of each IS acquired against the mid point of the ICAL. The Extracted Ion Current Profile (EICP) must be within a factor of two (-50 to +100%).
- ✓ Check concentration of target analytes if calibration range is exceeded.
- ✓ Check surrogate recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 QC limits.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. Note observations on the analysis log. When results arise to questionable result, e.g. inconsistency from the first analysis, consult the Supervisor for further action.

10.4.2.2. Properly fill up the analysis log.

10.4.3. Qualitative Identification

- The intensities of the characteristic ions maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
- Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. For manual integration refer to EMAX-DM01.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.<sup>4</sup>

10.4.3.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification<sup>5</sup> (TIC). Execute LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library. Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting following the guidelines below.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within + 20%. Example: for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.

<sup>4</sup> SW846 Method 8260B, Section 7.6.1.4

<sup>5</sup> Library search is performed only when indicated in the PSR.

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- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting analytes.
- Ions present in the reference spectrum but not present in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting analytes. Data system library reduction programs can sometimes create these discrepancies.

**10.4.3.2. Reporting TICs**

- If the library search produces a match at or above 85%, report the analyte.
- If the library search produces more than one analyte at or above 85%, report the first analyte (highest).
- If the library search produces no matches at or above 85%, the compound should be reported as unknown.

**10.4.4. Quantitation**

10.4.4.1. Apply the appropriate quantitation method (Section 10.3.3.6). Calculate the concentration of any positively identified target analyte using Eq.-10.5.3. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

**10.4.5. Manual Integration**

10.4.5.1. Refer to EMAX-DM01, Manual Integration Section.

**10.4.6. Dealing with Carryover**

10.4.6.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.6.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.6.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.6.4. To clean-up the autosampler purge line consider purging a 25 ml or 5 ml sample spiked with 100 µl of methanol and let it run like a blank sample. If improved result is noted repeat this process until no evidence of contamination is observed. Otherwise inform the Supervisor for further instruction.

**10.5. Calculations****10.5.1. Initial Calibration****10.5.1.1. Calculate for the Relative Response Factor (RRF)**

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$$RRF = \frac{A_X C_{IS}}{A_{IS} C_X} \quad \text{Eq.-10.5.1.1}$$

where:

- $A_X$  – Area of characteristic ion for the compound being measured  
 $A_{IS}$  – Area of characteristic ion for the specific internal standard  
 $C_X$  – Concentration of the compound being measured  
 $C_{IS}$  – Concentration of the specific internal standard

**10.5.1.2. Calculate for the Average Relative Response Factor (RRF<sub>m</sub>)**

$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq.-10.5.1.2}$$

where:

- $\sum RRF$  – Summation of response factors  
 $n$  – Number of measurements

**10.5.1.3. Calculate for Least Square Linear Regression**

$$y = ax + b \quad \text{Eq.-10.5.1.3}$$

where:

- $y$  – Response ratio ( $A_X/A_{IS}$ )  
 $x$  – Amount ratio ( $C_X/C_{IS}$ )  
 $a$  – x1 = slope of the line  

$$a = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

where:

- $\bar{x}$  = average of amount ratios  
 $\bar{y}$  = average of response ratios  
 $b$  – x0 = intercept of the line  

$$b = \bar{y} - a * \bar{x}$$

**10.5.1.4. Calculate for Inverse Weighting Factor**

$$y = ax + b \quad \text{Eq.-10.5.1.4}$$

where:

- $y$  – Response ratio ( $A_X/A_{IS}$ )  
 $x$  – Amount ratio ( $C_X/C_{IS}$ )  
 $a$  – x1 = slope of the line  

$$a = \frac{\sum (x - x_a)(y - y_a)}{\sum (x - x_a)^2}$$

where:

$$x_a = \frac{\sum [x(1/x)]}{\sum (1/x)}$$

$$y_a = \frac{\sum [y(1/x)]}{\sum (1/x)}$$

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$$\text{or } x_a = \frac{\sum [x(1/x^2)]}{\sum (1/x^2)}$$

$$y_a = \frac{\sum [y(1/x^2)]}{\sum (1/x^2)}$$

$$b - x_0 = \text{intercept of the line}$$

$$b = y_a - a * x_a$$

## 10.5.1.5. Calculate Inverse Quadratic

$$y = ax^2 + bx + c$$

where:

$$y - \text{Resp\_Ratio} = x_0 + x_1 * \text{Amt\_Ratio} + x_2 * (\text{Amt\_Ratio})^2$$

$$x - \text{Amt\_Ratio}$$

$$c - x_0 = \text{Det } 0 / \text{Det } b$$

$$b - x_1 = \text{Det } 1 / \text{Det } b$$

$$a - x_2 = \text{Det } 2 / \text{Det } b$$

$$W_i = \frac{\frac{1}{x_i}}{\sum_{i=1}^n \frac{1}{x_i}}$$

where:

$$X_i = \text{amount ratio} = \text{Conc of Std} / \text{Conc of IS}$$

$$Y_i = \text{response ratio} = \text{Resp of Std} / \text{Resp of IS}$$

$$W_i = 1/X_i / \text{SUM}(1/X_i)$$

$$\langle X \rangle = \text{SUM}(W_i * X_i)$$

$$\langle Y \rangle = \text{SUM}(W_i * Y_i)$$

$$\langle XX \rangle = \text{SUM}(W_i * (X_i)^2)$$

$$\langle XXX \rangle = \text{SUM}(W_i * (X_i)^3)$$

$$\langle XXXX \rangle = \text{SUM}(W_i * (X_i)^4)$$

$$\langle YY \rangle = \text{SUM}(W_i * (Y_i)^2)$$

$$\langle XY \rangle = \text{SUM}(W_i * X_i * Y_i)$$

$$\langle XXY \rangle = \text{SUM}(W_i * (X_i)^2 * Y_i)$$

$$\langle Yd2 \rangle = \text{SUM}((Y_i - \langle Y \rangle)^2 * W_i)$$

$$Ye = x_0 + x_1 * X_i + x_2 * X_i^2 - \langle Y \rangle$$

$$\langle Ye2 \rangle = \text{SUM}(Ye^2 * W_i)$$

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Det b	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

Det 0	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<Y>	<XY>	<XXY>

Det 1	1	<X>	<XX>
	<Y>	<XY>	<XXY>
	<XX>	<XXX>	<XXXX>

Det 2	<Y>	<XY>	<XXY>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

$$r^2 = \frac{<Ye2>}{<Yd2>}$$

$$ccf2 = (r^2)^{1/2}$$

## 10.5.1.6. Calculate the Standard Deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq.-10.5.1.6}$$

where:

- $x_i$  – Result at  $i^{\text{th}}$  measurement
- $\bar{x}$  – Mean of the  $n$  measurements
- $n$  – Number of measurements

## 10.5.1.7. Calculate the % relative standard deviation (%RSD)

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq.-10.5.1.7}$$

where:

- $SD$  – Standard deviation
- $RRF_m$  – Average response factor

## 10.5.1.8. Calculate the relative retention time (RRT)

$$RRT = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the Internal Standard}} \quad \text{Eq.-10.5.1.8}$$

10.5.2. Calibration Check/Continuing Calibration

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$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq.-10.5.2.1}$$

where:

- $RRF_c$  – Response factor from continuing calibration standard  
 $RRF_m$  – Average response factor

10.5.2.2. **% Drift**

$$\%Drift = \frac{[found\ Conc. - true\ Conc.]}{true\ Conc.} * 100\% \quad \text{Eq.-10.5.2.2}$$

10.5.3. **Calculation of Sample Concentration (Water and Soil/Sediment Samples)**

10.5.3.1. When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.5.3.2. **Water Samples**

$$Concentration\ (ug/L) = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)} \times DF \quad \text{Eq.-10.5.3.2}$$

where:

- $A_x$  – Area of characteristic ion for the compound to be measured  
 $I_s$  – Concentration of internal standard added in  $\mu\text{g/L}$   
 $A_{is}$  – Area of characteristic ion for the internal standard  
 $RRF_m$  – Average response factor  
 $DF$  – Dilution factor =  $\frac{\text{purge volume in ml (5 ml or 25 ml)}}{\text{sample amount in ml}}$

10.5.3.3. **Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)(DW)} \times DF \quad \text{Eq.-10.5.3.3}$$

where:

- $A_x$  – Area of characteristic ion for the compound to be measured  
 $I_s$  – Concentration of internal standard added in  $\mu\text{g/L}$   
 $A_{is}$  – Area of characteristic ion for the internal standard  
 $RRF_m$  – Average response factor  
 $DF$  – Dilution factor =  $\frac{5\ \text{g}}{(\text{sample amount in g})}$   
 $DW$  – % solid =  $\frac{100 - \%moisture}{100}$

10.5.3.4. **Extracted Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)(DW)} \times DF \quad \text{Eq.-10.5.3.4}$$

where:

- $A_x$  – Area of characteristic ion for the compound to be measured

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$$\begin{aligned}
 I_s & - \text{Concentration of internal standard added in } \mu\text{g/L} \\
 A_{is} & - \text{Area of characteristic ion for the internal standard} \\
 RRF_m & - \text{Average response factor} \\
 DF & - \text{Dilution factor} \\
 DW & - \text{\% solid} = \frac{100 - \% \text{moisture}}{100} \\
 & \quad \frac{(purged \text{ volume in } \mu\text{L})(5 \text{ g})}{(extract \text{ aliquot in } \mu\text{L})(sample \text{ amount in g})}
 \end{aligned}$$

10.5.4. Alternatively, the regression line (area ratio of  $A_x/A_{is}$  versus concentration using first degree) fitted to the initial calibration may be used for determination of the sample concentration when RSD of the analyte is greater than 15% (Section 10.3.3.6) .

10.5.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.5.5.1. The area  $A_x$  and  $A_{is}$  are derived from total ion chromatogram.  $A_{is}$  refers to the closest internal standard (IS) free of interference.

10.5.5.2. RRF of the TIC is 1.

10.5.6. Method Proficiency

10.5.6.1. **Percent Recovery**

$$\% \text{ Recovery} = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.5.6.1}$$

where:

- $C_f$  - Concentration found
- $C$  - Concentration of sample (use 0 for LCS)
- $C_s$  - Concentration of spike

10.5.6.2. **Relative Percent Difference (RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.6.2}$$

where:

- $C_1$  - Measured concentration of the first sample aliquot
- $C_2$  - Measured concentration of the second sample aliquot

10.6. **Data Reduction**

10.6.1. Make a copy of the analysis log.

10.6.2. Print a copy of the sample weight log (if any).

10.6.3. Highlight the data to be reported.

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- 10.6.4. Print a copy of the raw data and the QC report.
- 10.6.5. Collate the reportable raw data separating the QC results from the sample results.
- 10.6.6. Keep all other data generated with the analytical folder marked with "For record only".

**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WDB1C.exe.
- 10.7.2. Generate the sample results using F1NV3C.exe or F1NV3C4.exe.
- 10.7.3. Generate the QC summary using QCV3CN.exe or QCV3CN4.exe.
- 10.7.4. Generate the Instrument Performance Check (ICAL and DCC) using F5VOA.exe.
- 10.7.5. Generate the IS and RT summary using F8VC.exe.
- 10.7.6. Generate Lab Chronicle using LABCHRN1.exe
- 10.7.7. Generate Case Narrative using CN1.exe
- 10.7.8. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
  - 10.7.8.1. Case Narrative
  - 10.7.8.2. Lab Chronicle
  - 10.7.8.3. Sample Results
  - 10.7.8.4. Method Blank Results
  - 10.7.8.5. LCS/LCSD Summary
  - 10.7.8.6. MS/MSD Summary
  - 10.7.8.7. Instrument Performance Check (ICAL)
  - 10.7.8.8. ICAL Summary
  - 10.7.8.9. ICV Summary
  - 10.7.8.10. Instrument Performance Check (DCC)
  - 10.7.8.11. IS and RT Summary
  - 10.7.8.12. DCC Summary
  - 10.7.8.13. Analysis Log
  - 10.7.8.14. Sample Weight Log (if any)
  - 10.7.8.15. Non-Conformance Report (If any)

**10.8. Data Review**

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
  - 10.8.1.1. If any of the checkpoints below indicates a problem, re-analysis is required.
    - ✓ Check internal standard area. They should be within -50 to +100% of ICAL midpoint to be acceptable, otherwise follow PSR.

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- ✓ Check retention time of each IS to the ICAL midpoint. They should be within  $\pm 30$  seconds to be acceptable, otherwise follow PSR.
- ✓ Check surrogate recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check concentration of target analytes if calibration range is exceeded.

10.8.1.2. Review the attached logs that they are properly filled.

10.8.1.3. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.4. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review.

#### 10.9. Preventive Maintenance

10.9.1. Perform instrument routine preventive maintenance and record on instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2. The table below list suggested routine maintenance schedule.

Task	Every Day	Every Week	Every Month	Every 3 Months	Every 6 Months	As Needed
Tune Check	✓					
Check gas cylinders pressure	✓					
Check the foreline pump oil level		✓				
Check the calibration vial						✓
Check and if necessary, change injection port liners, septa and O-rings.					✓	
Replace the foreline pump oil					✓	
Replace the diffusion pump fluid					✓	
Replace the traps and filters					✓	
Clean the ion source						✓
Change the carrier gas trap(s) and purifier						✓
Replace column						✓
AutoTune the MSD						✓
Replace the worn out parts						✓

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**11.0 QUALITY CONTROL****11.1. Analytical Batch QC**

- 11.1.1. Perform tune check to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to calibration and check for any contamination.
- 11.1.2. Perform initial calibration (ICAL) to establish a calibration curve for the quantification of the analytes of interest.
- 11.1.3. Establish retention time window position for each analyte every after ICAL for proper qualitative identification.
- 11.1.4. Perform initial continuing calibration verification (ICV) every after ICAL to verify accuracy of ICAL.
- 11.1.5. Perform continuing calibration verification (CCV) every 12 hours to verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.
- 11.1.6. Evaluate relative retention time for each analytes in every sample to be within  $\pm 0.06$  RRT units.
- 11.1.7. Verify internal standard (IS) for quantitative accuracy and that its Retention time is within  $\pm 30$  seconds from retention time of the midpoint standard in the ICAL and EICP area is within -50% to +100% of ICAL midpoint standard.
- 11.1.8. Evaluate surrogate recovery to monitor instrument response on every sample.

**11.2. Preparation Batch QC**

- 11.2.1. Reagent water used for IB shall be of the same source for all QC samples and sample dilutions.
- 11.2.2. Analyze MB, LCS, MS/MSD and < 20 field samples.
- 11.2.3. Solvents and reagents must undergo quality control check prior to its use. Refer to EMAX-QC01 for details.
- 11.2.4. Properly treat lab wares used in the sample preparation as specified in EMAX-QC07.

**11.3. Method QC**

- 11.3.1. All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.
  - 11.3.2. All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.
- 11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

**12.0 CORRECTIVE ACTION**

12.1. Corrective action for each Quality Control procedure is summarized in Appendix 1.

**12.2. Analytical Batch QC**

- 12.2.1. Tune Check – If tune check is non-compliant consider the following suggestion to correct the problem:
  - Check the abundance of mass 95 and 174. If it is significantly less than previous tune checks, it is indicative of insufficient amount of BFB injected. Probable causes are:

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improper spiking, leaks, standard degradation or low vacuum system. Repeat tune check ensuring that BFB was properly spiked or rule out leaks, prepare a fresh BFB standard and repeat the tune check.

- If problem persist, re-tune the instrument and repeat tune check.
- If problem is unresolved, inform the supervisor for further action.

**12.2.2. Initial Calibration**

12.2.2.1. If the %RSD is out of acceptance criteria, consider the following suggestions to correct the problem.

- If one of the standards returns a bias low or bias high on all of the analytes then that point is considered an out-liner. Prepare a standard at that ICAL point and re-analyze.
- If the highest ICAL point appears to be saturated, drop the highest point.
- If the lowest point returns a bias low or bias high response or the peaks are not distinct and sharp, drop the lowest point.

*Note : The lowest calibration point identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.*

12.2.2.2. If instrument problem is suspected, consider the following suggestion to correct the problem:

- Check the connection and make sure they are air tight and perform maintenance as needed.
- Check the gas flow.
- Re-tune the MS.
- Prepare a fresh standard and repeat calibration.
- Clean the MS source and repeat calibration.
- If problem is unresolved, inform the supervisor for further action.

12.2.3. **Initial Calibration Verification (ICV)** – If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Re-analyze ICV to rule out poor purge.
- If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If problem is unresolved, inform the supervisor for further action.

12.2.4. **Daily Calibration Check (DCC)** – If DCC is non-compliant consider the following suggestions to correct the problem:

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- If majority of the analyte response are low and no evidence of leak in the system is apparent, it is indicative of a bad purge or leak in the vial. Re-analyze DCC.
- If problem persist, rule out standard degradation. Prepare a fresh standard and repeat DCC.
- Otherwise execute instrument maintenance and perform ICAL.

12.2.5. Instrument Blank – If instrument blank is non-compliant, consider the following suggestions to correct the problem:

- If trace level of THMs is observed, it is indicative that water filters need replacement. Otherwise, bake the trap at the manufacturer's recommended temperature for about 30 minutes.
- If contamination is high, flush the sample line with methanol and replace the trap.
- If problem is unresolved, inform the supervisor for further action.

**12.3. Preparation Batch QC**

12.3.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.3.2. If MB is non-compliant, consider the suggestions as described in Instrument Blank.

12.3.3. If LCS is non-compliant, consider the following suggestions to correct the problem:

- If result is bias low or high, prepare a fresh standard and re-analyze LCS and the associated samples.
- If problem is unresolved, inform the supervisor for further advice.

12.3.4. If MS is non-compliant consider the following suggestion to correct the problem:

- Check the standard log and analytical log and verify that the spike amount value used for calculation is correct.
- If LCS is within acceptance criteria then and the right amount of spike amount used for calculation is correct, then it is indicative of matrix interference. Discuss the probable matrix interference in the case narrative.

12.4. Discuss water samples that are labeled preserved having a pH  $\geq 2$  and/or residual chlorine  $\geq 5$  mg/L in the case narrative.

12.5. A Non-Conformance Report (NCR) is required when the following circumstances occur.

- Anomalies other than specified in Appendix 1, is observed.
- Sample is out of technical holding time.

12.5.1. Refer to EMAX-QA08 for NCR details.

**13.0 POLLUTION PREVENTION**

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

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**14.0 WASTE MANAGEMENT**

- 14.1. No samples shall be dumped on the laboratory sink.
- 14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.
- 14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.
- 14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

- 15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
  - 15.1.2.1. **Preparation Batch** - is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
  - 15.1.2.2. **Analytical batch** - is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.3. Detection Limit (DL) – is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.
- 15.1.4. Limit of Detection (LOD) – is defined as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative result rate (Type II error) is 1 %.
- 15.1.5. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- 15.1.6. Safety Data Sheet (SDS) – is written information concerning a chemical physical properties, toxicity, health hazards, fire hazard and reactivity data including storage, spill and handling precautions.
- 15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

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- 15.1.9. Carry-over – are contaminants retained in the instrument/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).
- 15.1.10. Calibration Check Compounds (CCC) – evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.
- 15.1.11. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.12. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.13. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.14. Lab Control Sample Duplicate (LCS D) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.15. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.16. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.17. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.18. Matrix – is a component or form of a sample.
- 15.1.19. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.20. Matrix Spike Duplicate (MS D) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.21. Response Factor – is the ratio of the peak area of the target compound in the sample or sample extract.
- 15.1.22. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.
- 15.1.23. SPCC – System performance check compounds are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.
- 15.1.24. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.
- 15.1.25. Reagent Soil – organic-free Ottawa sand or equivalent.
- 15.2. **Application of EMAX QC Procedures**

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15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Volatile analysis by GC/MS. The standard analyte list and RL are presented in Tables 6 & 7. In instances where there is a project or program QAPP, the requirements given in the project takes precedence over this SOP.

**15.3. Department of Defense (DoD) and Department of Energy (DoE) Projects**

15.3.1. Samples from DoD and DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

**16.0 REFERENCES**

- 16.1. U.S. EPA Method 8260B; SW846, as updated
- 16.2. EMAX Quality Systems Manual, as updated

**17.0 APPENDICES****17.1. Tables**

- 17.1.1. Table 1 Initial Calibration Intermediate Standards Preparation
- 17.1.2. Table 2 Initial Calibration Verification/LCS/MS/MSD Intermediate Standards Preparation
- 17.1.3. Table 3 Surrogate/Internal Standards Preparation
- 17.1.4. Table 4 Tuning Solution Standards Preparation
- 17.1.5. Table 5 BFB Key Ion Abundance Criteria
- 17.1.6. Table 6 Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards, Detection Limits for 5 ml Purge
- 17.1.7. Table 7 Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards, Detection Limits for 25 ml Purge

**17.2. Figures**

- 17.2.1. Figure 1 Peak Evaluation Techniques
- 17.2.2. Figure 2 Typical Chromatogram
- 17.2.3. Figure 3 Typical ICAL Summary
- 17.2.4. Figure 4 Typical Instrument Performance Check (Tuning)
- 17.2.5. Figure 5 Typical Instrument Performance Check (Tuning) Summary
- 17.2.6. Figure 6 Typical Internal Standard Area and Retention Time Summary
- 17.2.7. Figure 7 Typical Sample Result Summary
- 17.2.8. Figure 8 Typical LCS/LCSD Summary
- 17.2.9. Figure 9 Typical MS/MSD Summary
- 17.2.10. Figure 10 Typical Case Narrative

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**17.3. Appendices**

- 17.3.1. Appendix 1 Summary of Quality Control Procedures
- 17.3.2. Appendix 2 Demonstration of Capability for 25 ml
- 17.3.3. Appendix 3 Demonstration of Capability for 5 ml
- 17.3.4. Appendix 4 Demonstration of Capability for 5 g

**17.4. Forms**

- 17.4.1. 8260FS Sample Preparation Log
- 17.4.2. 8260FA Analytical Run Log
- 17.4.3. 8260FM Instrument Maintenance Log

**Table 1: INITIAL CALIBRATION INTERMEDIATE STANDARDS PREPARATION**

ICAL/DCC Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
I	1-Chlorohexane	AccuStandard	2000	50	2	50
	2-Chloroethylvinylether	CPI	2000	50	2	50
	Oxygenate Gasoline Additive	AccuStandard	2000-10000	50	2	50 - 250
	Custom VOA Mix	CPI	2000, 20000, 40000	50	2	50, 500, 1000
II	VOC Gas Mix	Ultra Scientific	2000	250	2	250
	Vinyl Acetate	CPI	2500	200	2	250
III	Carbon Disulfide	CPI	5000	100	2	250
IV	VOA Calibration Mix 1	Restek	5000	100	2	250
	Acrolein / Acrylonitrile	AccuStandard	5000	100	2	250

**Table 2: INITIAL CALIBRATION VERIFICATION/LCS/MS/MSD INTERMEDIATE STANDARDS PREPARATION**

ICV / LCS / MS Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (µl)	Final Vol. (ml)	
I	1-Chlorohexane	Ultra Scientific	1000	100	2	50
	2-Chloroethylvinylether	AccuStandard	2000	50	2	50
	California Oxygenate Mix	Restek	2000 - 10000	50	2	50 – 250
	Custom 8260 Mega Mix	Restek	2000, 20000, 40000	50	2	50, 500, 1000
II	Volatile Organic Cpds Mix 6	Supelco	2000	250	2	250
	Vinyl Acetate	Restek	2000	250	2	250
III	Carbon Disulfide Solution	Ultra scientific	5000	20	2	50
IV	TCL Volatile Mix 1	Supelco	2000	250	2	250
	Acrolein / Acrylonitrile	Ultra Scientific	2000	250	2	250

**Table 3: SURROGATE / INTERNAL STANDARDS PREPARATION**

Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (μl)	Final Vol. (ml)	
Surrogate	8260 Surrogate Mix	Restek	2500	200	2	250
Internal Standard	Custom 8260 Internal Standard Mix, 3-30	CPI	2500	200	2	250

**Table 4: TUNING SOLUTION STANDARDS PREPARATION**

BFB Intermediate Standard	Stock Standard			Preparation (Solvent: Methanol)		Final Conc. (mg/L)
	Standard Name	Source	Conc. (mg/L)	Aliquot (μl)	Final Vol. (ml)	
Tuning Compound	BFB	Restek	5000	20	2	50

**Table 5: BFB KEY ION ABUNDANCE CRITERIA**

M/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

**Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE**

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8			DL	LOD	LOQ	DL	LOD	LOQ
1,1,1,2-Tetrachloroethane	131	133, 119, 117	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,1-Trichloroethane	97	99, 61	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,2,2-Tetrachloroethane	83	85	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1,2-Trichloro-1,2,2-trifluoroethane	151	153	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,1,2-Trichloroethane	97	83, 85, 99	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloroethane	63	65, 83	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloroethene	61	63, 96	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,1-Dichloropropene	110	112	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2,3-Trichlorobenzene	180	182, 145	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
1,2,3-Trichloropropane	110	61, 77	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,2,4-Trichlorobenzene	180	182, 145	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
1,2,4-Trimethylbenzene	105	120	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.55	1	5
1,2-Dibromo-3-chloropropane	157	155, 75	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,2-Dibromoethane	107	109	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichloroethane	62	64	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,2-Dichloropropane	63	41, 76	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,3,5-Trimethylbenzene	105	120, 119	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.59	1	5
1,3-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.52	1	5
1,3-Dichloropropane	76	78	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1,4-Dichlorobenzene	146	111, 148	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
1-Chlorohexane	91	93, 55, 56	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.58	1	5
2,2-Dichloropropane	77	97, 79	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
2-Butanone (MEK)	43	72	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
2-Chloroethyl vinyl ether	63	65, 106	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
2-Chlorotoluene	91	126	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.82	1	5
2-Hexanone (MBK)	43	58, 100	IS2	Sur3	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.86	5	10
4-Chlorotoluene	91	126	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.61	1	5	0.67	1	5
4-Methyl-2-pentanone (MIBK)	43	58, 85, 100	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.75	5	10
Acetone	43	58, 42	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	3.06	5	10
Acrolein	56	55	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
Acrylonitrile	53	52, 51	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	2.5	5	10	2.5	5	10
Benzene	78	77, 52	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromobenzene	156	77, 158	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromochloromethane	49	128, 130	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromodichloromethane	83	85	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Bromoform	173	171, 175	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Bromomethane	94	96	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1.1	2	5	1.81	2	5

**Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE**

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8			DL	LOD	LOQ	DL	LOD	LOQ
Carbon disulfide	76	78	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Carbon tetrachloride	119	117	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.54	1	5
Chlorobenzene	112	51, 77, 114	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Chloroethane	64	49, 66	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Chloroform	83	85, 47	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Chloromethane	50	52	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1.02	2	5
cis-1,2-Dichloroethene	96	61, 98	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
cis-1,3-Dichloropropene	75	77, 39, 110	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dibromochloromethane	129	127	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dibromomethane	93	95, 174	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Dichlorodifluoromethane	85	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1.3	2	5	1.16	2	5
Dichlorofluoromethane	67	69	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Diisopropyl ether (DIPE)	45	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Ethyl Methacrylate	69	99, 41	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Ethylbenzene	91	106	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Ethyl-tert-butyl ether (ETBE)	59	87	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Hexachlorobutadiene	225	223, 227	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Iodomethane	142	127	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Isopropylbenzene	105	120, 79, 103	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.64	1	5
m/p-Xylenes	91	106	IS2	Sur3	4	10	20	40	100	160	200	400	100	100	1	2	10	1	2	10
Methylene chloride	49	84, 86	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	1	2	5
Methyl-t-butyl ether (MTBE)	73	57	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
Naphthalene	128	127	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
n-Butylbenzene	91	92, 134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.73	1	5	0.7	1	5
n-Propylbenzene	91	65, 120	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.51	1	5	0.65	1	5
o-Xylene	91	106	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
p-Isopropyltoluene	119	91, 134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.56	1	5	0.62	1	5
sec-Butylbenzene	105	134	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.67	1	5
Styrene	104	78, 103	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
tert-Amylmethyl ether (TAME)	87	55, 73	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
tert-Butyl alcohol (TBA)	59	41	IS1	Sur0/1	10	25	50	100	250	400	500	1000	250	250	7.1	10	25	9.18	10	20
tert-Butylbenzene	134	91, 119	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.62	1	5
Tetrachloroethene	164	129, 131, 166	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.52	1	5	0.5	1	5
Toluene	91	92	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,2-Dichloroethene	61	96, 98	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,3-Dichloropropene	75	77, 39	IS2	Sur3	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5
trans-1,4-Dichloro-2-butene	53	88	IS3	Sur2	2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Trichloroethene	130	97, 132, 95	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.5	1	5	0.5	1	5

**Table 6: TYPICAL TARGET ANALYTE LIST FOR 5-ml PURGE**

ANALYTES	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8			DL	LOD	LOQ	DL	LOD	LOQ
Trichlorofluoromethane	101	103	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.86	1	5	1.06	2	5
Vinyl acetate	43	86	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	1	2	5	1.26	2	5
Vinyl chloride	62	64	IS1	Sur0/1	2	5	10	20	50	80	100	200	50	50	0.61	1	5	1	2	5
Dibromofluoromethane (Sur0)	111	113, 192	IS1		2	5	10	20	50	80	100	200	50	50	1	2	5	0.5	1	5
1,2-Dichloroethane-d4 (Sur1)	65	102	IS1		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
4-Bromofluorobenzene (Sur2)	95	174, 176	IS3		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
Toluene-D8 (Sur3)	98	100	IS2		2	5	10	20	50	80	100	200	50	50	1	2	5	1	2	5
1,4-Difluorobenzene (IS1)	114	88																		
Chlorobenzene-d5 (IS2)	117	82, 119																		
1,2-Dichlorobenzene-d4 (IS3)	152	150																		

Note: Since, retention time of Dibromofluoromethane (Sur0) and 1,2-Dichloroethane-d4 (Sur1) is too close (~43 sec) hence, Dibromofluoromethane (Sur0) is only used when required by the project.

**Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE**

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
1,1,1,2-Tetrachloroethane	131	133, 119, 117	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1,1-Trichloroethane	97	99, 61	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1,2,2-Tetrachloroethane	83	85	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,1,2-Trichloro-1,2,2-trifluoroethane	151	153	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.17	0.3	0.5
1,1,2-Trichloroethane	97	83, 85, 99	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloroethane	63	65, 83	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloroethene	61	63, 96	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,1-Dichloropropene	110	112	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2,3-Trichlorobenzene	180	182, 145	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
1,2,3-Trichloropropane	110	61, 77	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
1,2,4-Trichlorobenzene	180	182, 145	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
1,2,4-Trimethylbenzene	105	120	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,2-Dibromo-3-chloropropane	157	155, 75	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
1,2-Dibromoethane	107	109	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichloroethane	62	64	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,2-Dichloropropane	63	41, 76	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,3,5-Trimethylbenzene	105	120, 119	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
1,3-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
1,3-Dichloropropane	76	78	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1,4-Dichlorobenzene	146	111, 148	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
1-Chlorohexane	91	93, 55, 56	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.14	0.2	0.5
2,2-Dichloropropane	77	97, 79	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.16	0.2	0.5
2-Butanone (MEK)	43	72	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2	4	10
2-Chloroethyl vinyl ether	63	65, 106	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
2-Chlorotoluene	91	126	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.12	0.2	0.5
2-Hexanone (MBK)	43	58, 100	IS2	Sur3	1.5	2.5	5	10	25	50	100	150	200	50	50	2.3	4	5
4-Chlorotoluene	91	126	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
4-Methyl-2-pentanone (MIBK)	43	58, 85, 100	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.2	4	5
Acetone	43	58, 42	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.6	5	10
Acrolein	56	55	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.5	5	10
Acrylonitrile	53	52, 51	IS1	Sur0/1	1.5	2.5	5	10	25	50	100	150	200	50	50	2.5	5	10
Benzene	78	77, 52	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5

**Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE**

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
Bromobenzene	156	77, 158	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Bromochloromethane	49	128, 130	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Bromodichloromethane	83	85	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Bromoform	173	171, 175	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Bromomethane	94	96	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.16	0.3	0.5
Carbon disulfide	76	78	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Carbon tetrachloride	119	117	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chlorobenzene	112	51, 77, 114	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chloroethane	64	49, 66	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.27	0.3	0.5
Chloroform	83	85, 47	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Chloromethane	50	52	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
cis-1,2-Dichloroethene	96	61, 98	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
cis-1,3-Dichloropropene	75	77, 39, 110	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dibromochloromethane	129	127	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dibromomethane	93	95, 174	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Dichlorodifluoromethane	85	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Dichlorofluoromethane	67	69	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Diisopropyl ether (DIPE)	45	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Ethyl Methacrylate	69	99, 41	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Ethylbenzene	91	106	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Ethyl-tert-butyl ether (ETBE)	59	87	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
Hexachlorobutadiene	225	223, 227	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.22	0.3	0.5
Iodomethane	142	127	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Isopropylbenzene	105	120, 79, 103	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
m/p-Xylenes	91	106	IS2	Sur3	0.6	1	2	4	10	20	40	60	80	20	20	0.21	0.4	1
Methylene chloride	49	84, 86	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Methyl-t-butyl ether (MTBE)	73	57	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
Naphthalene	128	127	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
n-Butylbenzene	91	92, 134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.17	0.2	0.5
n-Propylbenzene	91	65, 120	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
o-Xylene	91	106	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
p-Isopropyltoluene	119	91, 134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.14	0.2	0.5
sec-Butylbenzene	105	134	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5

**Table 7: TYPICAL TARGET ANALYTE LIST FOR 25-ml PURGE**

Analytes	CHARACTERISTIC ION(S)		IS	SURR	ICAL ANALYTE CONCENTRATIONS (µg/L)									ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER (µg/L)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	9			DL	LOD	LOQ
Styrene	104	78, 103	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
tert-Amylmethyl ether (TAME)	87	55,73	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
tert-Butyl alcohol (TBA)	59	41	IS1	Sur0/1	1.5	2	5	10	25	50	100	150	200	50	50	2.5	5	10
tert-Butylbenzene	134	91, 119	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.13	0.2	0.5
Tetrachloroethene	164	129, 131, 166	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.2	0.5
Toluene	91	92	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
trans-1,2-Dichloroethene	61	96, 98	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
trans-1,3-Dichloropropene	75	77, 39	IS2	Sur3	0.3	0.5	1	2	5	10	20	30	40	10	10	0.11	0.2	0.5
trans-1,4-Dichloro-2-butene	53	88	IS3	Sur2	0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
Trichloroethene	130	97, 132, 95	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.1	0.2	0.5
Trichlorofluoromethane	101	103	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.15	0.3	0.5
Vinyl acetate	43	86	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.25	0.5	1
Vinyl chloride	62	64	IS1	Sur0/1	0.3	0.5	1	2	5	10	20	30	40	10	10	0.12	0.2	0.5
Dibromofluoromethane (Sur0)	111	113, 192	IS1		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
1,2-Dichloroethane-d4 (Sur1)	65	102	IS1		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
4-Bromofluorobenzene (Sur2)	95	174, 176	IS3		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
Toluene-D8 (Sur3)	98	100	IS2		0.3	0.5	1	2	5	10	20	30	40	10	10	0.5	1	2
1,4-Difluorobenzene (IS1)	114	88																
Chlorobenzene-d5 (IS2)	117	82, 119																
1,2-Dichlorobenzene-d4 (IS3)	152	150																

Note: Since retention time of Dibromofluoromethane (Sur0) and 1,2-Dichloroethane-d4 (Sur1) is too close (~43 sec) hence, Dibromofluoromethane (Sur0) is only used when required by the project.

**Figure 1:**

**PEAK EVALUATION TECHNIQUES**

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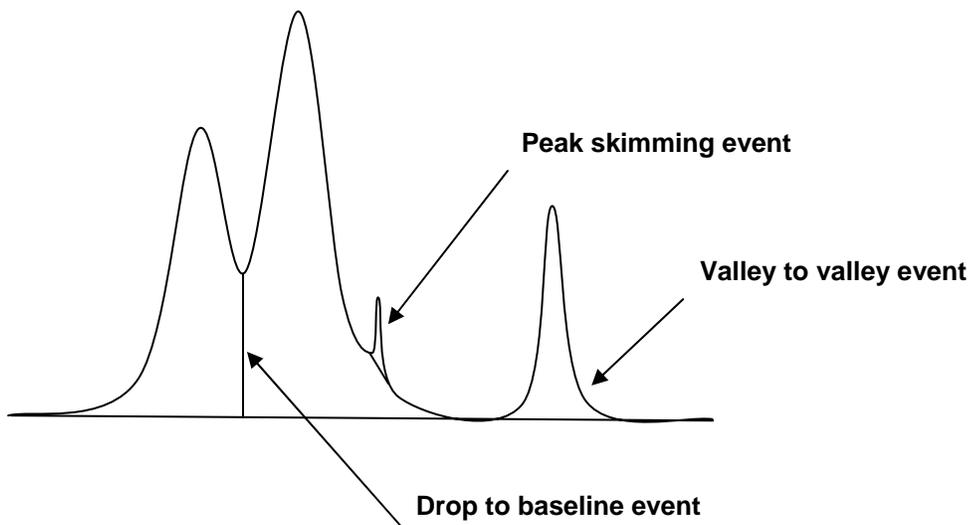


Figure 2:

TYPICAL CHROMATOGRAM

Quantitation Report

Data File : D:\HPCHEM\1\DATA\11E25\REC395.D  
Acq On : 25 May 2011 1:40 pm  
Sample : IVO67E2402 10ppb  
Misc : 10ppb 8260/50ppb KET-ACR-ACN-TBA  
MS Integration Params: LSCINT1.P  
Quant Time: May 25 16:32 2011

Vial: 2  
Operator: AS  
Inst : TO67  
Multiplr: 1.00

Quant Results File: VO67E24.RES

Method : D:\HPCHEM\1\METHODS\VO67E24.M (RTE Integrator)  
Title : METHOD 8260B 4.0  
Last Update : Wed May 25 16:26:28 2011  
Response via : Initial Calibration

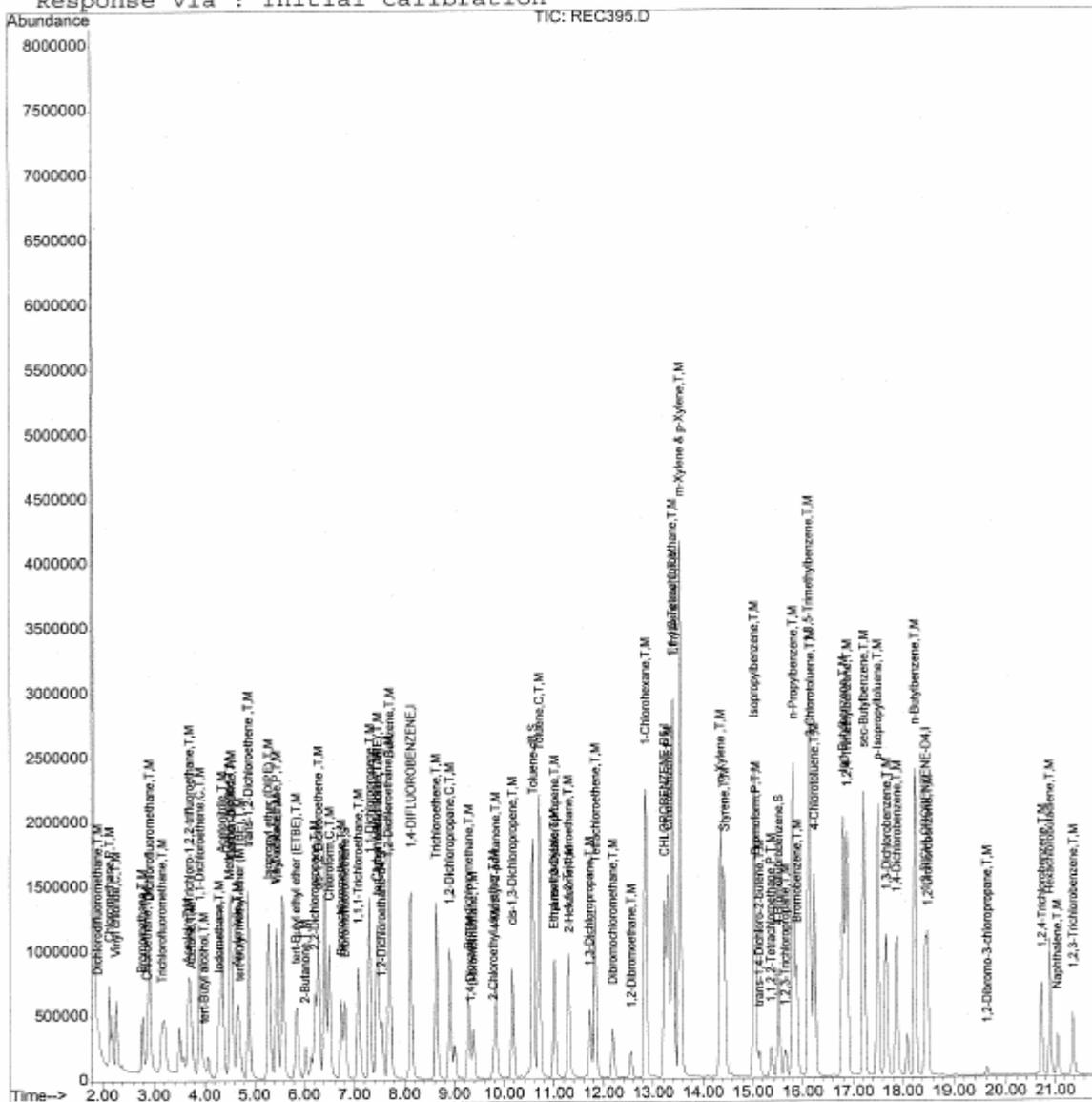


Figure 3:

TYPICAL ICAL SUMMARY

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Instrument ID :03  
Beginning DateTime :03/12/13 07:57  
Spike Units :PPB  
IC File :RCB023

Column Spec :ZB-624 ID :0.25MM  
Ending DateTime :03/12/13 12:58  
HPChem Method :V003C12

M_IDX	Parameters	.3 RCB018	.5 RCB019	1 RCB020	2 RCB021	5 RCB022	10 RCB023	20 RCB024	30 RCB025	50 RCB026	100 RCB027	Av_RRF	%_RSD	Av_Rt_M
1	1,4-DIFLUOROENZENE	1	1	1	1	1	1	1	1	1	1	1	0	13.1167
2	Chlorotrifluoroethylene											0.000	0.00	0.0000
3	Dichlorodifluoromethane	0.312	0.311	0.326	0.327	0.302	0.284	0.298	0.281	0.271	0.265	0.298	7.35	4.0527
4	Chloromethane	0.497	0.479	0.499	0.498	0.449	0.423	0.434	0.414	0.395	0.379	0.447	10.02	4.5516
5	Vinyl chloride	0.295	0.288	0.315	0.308	0.286	0.271	0.286	0.277	0.270	0.273	0.287	5.39	4.8866
6	2-Chloro-1,1,1-trifluoroethane											0.000	0.00	0.0000
7	Bromomethane	0.307	0.306	0.320	0.311	0.275	0.265	0.267	0.247	0.210		0.279	12.91	5.7735
8	Chloroethane	0.209	0.223	0.239	0.233	0.210	0.202	0.209	0.197	0.184	0.165	0.207	10.58	6.0466
9	Dichlorofluoromethane	0.670	0.653	0.680	0.650	0.648	0.592	0.632	0.621	0.635	0.625	0.641	3.96	6.5916
10	Trichlorofluoromethane	0.321	0.351	0.385	0.402	0.365	0.363	0.384	0.376	0.364	0.363	0.367	5.95	6.6749
5 11	Acrolein	0.012	0.010	0.011	0.011	0.012	0.013	0.013	0.013	0.013	0.013	0.012	9.23	7.7679
12	1,1,2-Trichloro-1,2,2-trifluoroethane	0.214	0.199	0.207	0.193	0.190	0.173	0.180	0.176	0.185	0.180	0.190	7.16	7.8901
13	1,1-Dichloroethene	0.613	0.612	0.620	0.579	0.570	0.534	0.548	0.523	0.543	0.514	0.566	6.97	7.9392
5 14	Acetone			0.045	0.035	0.032	0.031	0.029	0.027	0.029	0.028	0.032	18.18	8.1020
15	Iodomethane	0.553	0.519	0.566	0.525	0.512	0.471	0.490	0.476	0.484	0.486	0.508	6.41	8.3338
16	Carbon disulfide	1.259	1.220	1.291	1.367	1.204	1.194	1.218	1.179	1.183	1.151	1.227	5.20	8.4588
17	Methyl acetate											0.000	0.00	0.0000
18	Methylene chloride			0.548	0.484	0.450	0.419	0.422	0.397	0.422	0.401	0.443	11.51	9.0085
5 19	tert-Butyl alcohol	0.009	0.009	0.011	0.010	0.010	0.009	0.010	0.009	0.010	0.009	0.010	6.11	9.2093
20	tert-Butyl methyl ether (MTBE)	0.430	0.405	0.440	0.420	0.409	0.367	0.381	0.371	0.404	0.374	0.400	6.43	9.4267
21	trans-1,2-Dichloroethene	0.681	0.638	0.664	0.629	0.618	0.585	0.584	0.553	0.577	0.535	0.606	7.83	9.4834
5 22	Acrylonitrile	0.036	0.036	0.041	0.040	0.038	0.039	0.037	0.037	0.038	0.036	0.038	5.13	9.5161
23	Isopropyl ether (DIPE)	1.387	1.263	1.335	1.285	1.234	1.171	1.151	1.110	1.121	1.024	1.208	9.29	10.2472
24	1,1-Dichloroethane	0.768	0.739	0.749	0.730	0.706	0.676	0.668	0.644	0.653	0.605	0.694	7.62	10.2785
25	Vinyl acetate		0.332	0.283	0.324	0.337	0.355	0.379	0.413	0.347	0.317	0.343	10.92	10.3003
26	tert-Butyl ethyl ether (ETBE)	0.796	0.755	0.728	0.707	0.718	0.694	0.681	0.696	0.732	0.705	0.721	4.69	10.8667
27	2,2-Dichloropropane	0.395	0.372	0.378	0.353	0.339	0.345	0.323	0.310	0.300	0.263	0.338	11.78	11.2345
5 28	2-Butanone	0.083	0.072	0.068	0.069	0.064	0.064	0.055	0.056	0.058	0.053	0.064	14.51	11.2761
29	cis-1,2-Dichloroethene	0.469	0.440	0.442	0.430	0.415	0.405	0.384	0.379	0.390	0.365	0.412	8.05	11.2687
5 30	2-Butanol											0.000	0.00	0.0000
31	Bromochloromethane	0.307	0.309	0.315	0.309	0.309	0.274	0.277	0.276	0.288	0.257	0.292	6.89	11.6797
32	Tetrahydrofuran			0.048	0.045	0.037	0.034	0.033	0.036	0.036	0.034	0.038	13.93	11.7372
33	Chloroform	0.705	0.690	0.687	0.691	0.672	0.607	0.605	0.606	0.620	0.579	0.646	7.24	11.7705
34	Dibromofluoromethane	0.326	0.367	0.356	0.342	0.340	0.327	0.311	0.315	0.311	0.294	0.329	6.83	12.0326
35	1,1,1-Trichloroethane	0.523	0.515	0.500	0.484	0.512	0.471	0.477	0.472	0.470	0.435	0.486	5.50	12.0386
36	Cyclohexane											0.000	0.00	0.0000
37	1,1-Dichloropropene	0.209	0.194	0.192	0.189	0.183	0.178	0.181	0.182	0.174	0.159	0.184	7.23	12.2738
38	Carbon tetrachloride	0.467	0.452	0.446	0.423	0.441	0.427	0.429	0.426	0.426	0.403	0.434	4.16	12.2857
39	1,2-Dichloroethane-d4	0.270	0.266	0.275	0.269	0.258	0.251	0.225				0.259	6.60	12.5581
40	Benzene	1.724	1.622	1.501	1.421	1.497	1.348	1.382	1.392	1.393		1.476	8.50	12.6150
41	tert-Amyl methyl ether (TAME)	0.100	0.103	0.107	0.100	0.109	0.098	0.097	0.102	0.105	0.098	0.102	4.11	12.6595
42	1,2-Dichloroethane	0.299	0.308	0.290	0.294	0.303	0.287	0.279	0.277	0.287	0.256	0.288	5.20	12.6729
43	Trichloroethene	0.428	0.404	0.405	0.389	0.419	0.382	0.382	0.375	0.400	0.366	0.395	4.98	13.5291
44	Methylcyclohexane											0.000	0.00	0.0000
45	1,2-Dichloropropane	0.387	0.398	0.375	0.376	0.407	0.377	0.381	0.361	0.376	0.342	0.378	4.76	13.8776
20 46	1,4-Dioxane	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	9.47	14.0562
47	Dibromomethane	0.163	0.146	0.158	0.146	0.166	0.142	0.149	0.144	0.158	0.145	0.152	5.80	14.0935
48	Bromodichloromethane	0.438	0.407	0.437	0.429	0.470	0.419	0.416	0.430	0.448	0.413	0.431	4.33	14.2617

Figure 3 (cont.):

TYPICAL ICAL SUMMARY

49	2-Chloroethyl vinyl ether	-----	0.052	0.053	0.059	0.064	0.062	0.064	0.067	0.077	0.073	0.063	13.09	14.5606
50	cis-1,3-Dichloropropene	0.511	0.509	0.493	0.489	0.553	0.509	0.513	0.510	0.537	0.492	0.512	3.91	14.8291
51	4-Methyl-2-pentanone	0.182	0.169	0.170	0.164	0.180	0.168	0.154	0.160	0.170	0.144	0.166	6.86	14.9541
52	CHLOROBENZENE-D5	1	1	1	1	1	1	1	1	1	1	1	0	17.1073
53	Toluene-d8	1.471	1.484	1.407	1.468	1.417	1.414	1.426	1.350	1.404	-----	1.427	2.94	15.1712
54	Toluene	1.975	1.822	1.622	1.764	1.814	1.638	1.732	1.646	1.710	-----	1.747	6.46	15.2622
55	Ethyl methacrylate	0.274	0.248	0.237	0.247	0.287	0.268	0.263	0.270	0.286	0.266	0.264	6.15	15.5111
56	trans-1,3-Dichloropropene	0.383	0.368	0.358	0.376	0.420	0.392	0.406	0.404	0.429	0.403	0.394	5.77	15.5483
57	1,1,2-Trichloroethane	0.222	0.209	0.198	0.196	0.213	0.196	0.195	0.206	0.217	0.201	0.205	4.79	15.8267
58	Tetrachloroethene	0.438	0.389	0.368	0.385	0.382	0.355	0.371	0.366	0.383	0.352	0.379	6.42	15.9995
59	2-Hexanone	0.119	0.137	0.116	0.121	0.117	0.125	0.112	0.121	0.125	0.106	0.120	6.87	16.0248
60	1,3-Dichloropropane	0.416	0.397	0.392	0.417	0.411	0.384	0.385	0.389	0.415	0.377	0.398	3.79	16.0531
61	Dibromochloromethane	0.252	0.234	0.233	0.261	0.259	0.256	0.262	0.267	0.287	0.268	0.258	6.22	16.3941
62	1,2-Dibromoethane	0.207	0.208	0.195	0.197	0.212	0.193	0.202	0.205	0.224	0.208	0.205	4.47	16.5847
63	1-Chlorohexane	0.897	0.862	0.785	0.803	0.835	0.761	0.806	0.779	0.829	0.757	0.811	5.53	16.9495
64	Chlorobenzene	1.152	1.065	0.937	0.954	0.990	0.958	0.962	0.991	1.007	0.911	0.993	7.08	17.1475
65	Ethylbenzene	2.251	2.178	2.021	1.994	2.182	1.965	2.014	2.014	1.839	-----	2.051	6.29	17.1930
66	1,1,1,2-Tetrachloroethane	0.319	0.317	0.311	0.307	0.333	0.304	0.308	0.319	0.318	0.294	0.313	3.37	17.2264
67	m-Xylene & p-Xylene	1.713	1.626	1.515	1.469	1.497	1.439	1.490	1.387	-----	-----	1.517	6.90	17.3227
68	o-Xylene	1.694	1.653	1.478	1.477	1.519	1.428	1.437	1.428	1.440	-----	1.506	6.65	17.8581
69	Styrene	1.028	1.012	0.971	0.933	1.027	0.971	0.981	0.978	0.998	0.874	0.977	4.73	17.8787
70	1,2-DICHLOROBENZENE-D4	1	1	1	1	1	1	1	1	1	1	1	0	20.6245
71	Bromoform	0.330	0.326	0.329	0.333	0.339	0.334	0.362	0.385	0.425	0.401	0.356	9.92	18.2613
72	Isopropylbenzene	5.562	5.517	5.180	5.026	5.022	4.609	5.010	5.013	5.337	-----	5.142	5.76	18.2767
73	4-Bromofluorobenzene	1.603	1.546	1.384	1.356	1.335	1.258	1.308	1.333	1.419	1.324	1.386	7.83	18.5621
74	1,1,2,2-Tetrachloroethane	0.746	0.756	0.716	0.675	0.696	0.618	0.665	0.707	0.749	0.704	0.703	6.08	18.7036
75	trans-1,4-Dichloro-2-butene	0.187	0.180	0.176	0.186	0.198	0.177	0.197	0.205	0.223	0.201	0.193	7.69	18.7438
76	n-Propylbenzene	8.651	8.030	7.852	7.211	7.500	6.892	7.592	7.274	-----	-----	7.625	7.21	18.7857
77	1,2,3-Trichloropropane	0.177	0.170	0.160	0.153	0.158	0.140	0.147	0.149	0.157	0.138	0.155	7.93	18.8019
78	Bromobenzene	1.165	1.071	1.055	1.014	1.052	0.931	0.983	1.022	1.109	1.010	1.041	6.31	18.8227
79	1,3,5-Trimethylbenzene	4.694	4.553	4.332	4.080	4.256	3.733	4.106	4.107	4.355	3.299	4.151	9.67	18.9612
80	2-Chlorotoluene	5.150	4.552	4.413	4.224	4.372	3.923	4.126	4.108	4.517	3.691	4.308	9.28	18.9999
81	4-Chlorotoluene	4.441	3.866	3.719	3.646	3.838	3.506	3.604	3.728	4.047	3.296	3.769	8.31	19.1265
82	tert-Butylbenzene	1.011	0.999	0.944	0.893	0.936	0.837	0.894	0.889	0.955	0.865	0.922	6.14	19.4213
83	1,2,4-Trimethylbenzene	4.627	4.271	3.973	4.095	4.026	3.564	3.677	3.874	4.208	3.401	3.972	9.12	19.4794
84	sec-Butylbenzene	6.845	6.467	6.537	6.165	6.138	5.570	5.934	5.860	6.013	-----	6.170	6.32	19.6962
85	p-Isopropyltoluene	4.982	4.670	4.483	4.208	4.407	4.011	4.268	4.060	4.454	-----	4.394	6.93	19.8402
86	1,3-Dichlorobenzene	2.358	2.168	2.115	2.002	2.047	1.927	2.037	1.985	2.204	2.008	2.085	6.15	19.9961
87	1,4-Dichlorobenzene	2.379	2.019	1.958	1.903	1.942	1.756	1.873	1.837	2.027	1.854	1.955	8.73	20.1063
88	n-Butylbenzene	5.550	4.899	5.079	4.650	4.817	4.593	4.886	4.619	4.980	-----	4.897	6.06	20.3928
89	1,2-Dichlorobenzene	1.816	1.636	1.646	1.560	1.576	1.448	1.505	1.471	1.578	1.464	1.570	7.08	20.6528
90	1,2-Dibromo-3-chloropropane	0.063	0.069	0.073	0.072	0.076	0.068	0.073	0.073	0.078	0.074	0.072	6.04	21.7696
91	1,2,4-Trichlorobenzene	0.972	0.844	0.786	0.841	0.831	0.798	0.763	0.733	0.817	0.766	0.815	8.11	23.0070
92	Hexachlorobutadiene	-----	0.740	0.722	0.700	0.678	0.604	0.560	0.503	0.559	0.527	0.621	14.43	23.1360
93	Naphthalene	1.249	1.099	1.013	1.042	1.036	0.961	0.917	0.843	0.957	0.895	1.001	11.58	23.4983
94	1,2,3-Trichlorobenzene	0.700	0.631	0.619	0.614	0.617	0.556	0.499	0.460	0.505	0.476	0.568	14.08	23.9168

Spike Amount = Nominal Amount \* M  
Ave\_%RSD : 7.5                      Max\_%RSD : 18.2

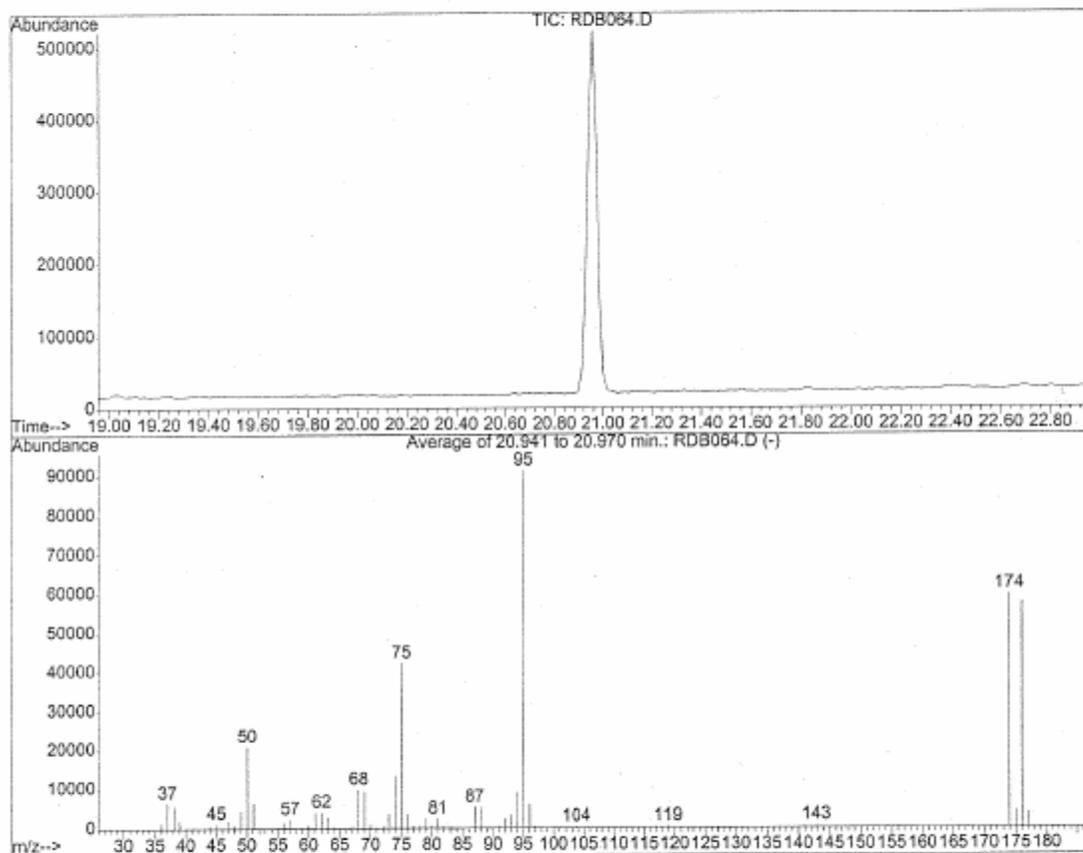
Use Least Square Linear Regression with weighting factor of inverse concentration for comps with %\_RSD > 15  
Resp\_Ratio = x0 + x1 \* Amt\_Ratio

IDX	Parameter	x0	x1	CCF
14	Acetone	0.00872	0.02772	0.9996

**Figure 4: TYPICAL INSTRUMENT PERFORMANCE CHECK (TUNING)**

BFB

Data File : D:\HPCHEM\1\DATA\11D07\RDB064.D Vial: 2  
 Acq On : 7 Apr 2011 7:43 pm Operator: MW  
 Sample : BFB03D04 Inst : TO03  
 Misc : T/CHECK Multiplr: 1.00  
 MS Integration Params: 524INT.P  
 Method : D:\HPCHEM\1\METHODS\VO03D07.M (RTE Integrator)  
 Title : METHOD 8260 25mL



AutoFind: Scans 1163, 1164, 1165; Background Corrected with Scan 1156

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	22.9	20916	PASS
75	95	30	60	46.4	42347	PASS
95	95	100	100	100.0	91301	PASS
96	95	5	9	6.6	6055	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	65.3	59619	PASS
175	174	5	9	7.4	4406	PASS
176	174	95	101	96.7	57635	PASS
177	176	5	9	6.7	3884	PASS



**Figure 6: TYPICAL INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY**

8A  
 VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name : EMAX INC.	Project : CLEAN WATER PROJECT
Lab Code : EMXT	SDG No. : YMNNN
Lab File ID : RDC168	Date Analyzed : 04/09/14
Instrument ID: 67	Time Analyzed : 18:54
GC Column : RTX502.2ID:0.25mm (mm)	Heated Purge : No

	IS1 (DFB)		IS2 (CBZ)		IS3 (DCB)	
	AREA #	RT #	AREA #	RT #	AREA #	RT #
12 HOUR STD	1321331	8.08	1022177	13.17	321226	18.40
UPPER LIMIT	2642662	8.58	2044354	13.67	642452	18.90
LOWER LIMIT	660666	7.58	511089	12.67	160613	17.90
SAMPLE ID						
1 VSTD010	1155410	8.07	908594	13.15	276831	18.38
2 MBLK1W	1259632	8.07	956984	13.15	261376	18.38
3 LCS1W	1216852	8.07	957974	13.15	290568	18.38
4 LCD1W	1262335	8.07	983468	13.15	300886	18.38
5 XXX-59GW001	1202358	8.07	934767	13.16	277693	18.38
6 XXX-59GW001MS	1216852	8.07	957974	13.17	290568	18.38
7 XXX-59GW001MSD	1262335	8.07	983468	13.17	300886	18.38

IS1 (DFB) = 1,4-Difluorobenzene  
 IS2 (CBZ) = Chlorobenzene-d5  
 IS3 (DCB) = 1,2-Dichlorobenzene-d4

AREA UPPER LIMIT = + 100% of internal standard area  
 AREA LOWER LIMIT = - 50% of internal standard area  
 RT UPPER LIMIT = + 0.5 minutes (30 sec) of internal standard RT  
 RT LOWER LIMIT = - 0.5 minutes (30 sec) of internal standard RT

# Column used to flag internal standard area values with an asterisk  
 \* Values outside of QC limits.

Figure 7:

TYPICAL SAMPLE RESULT SUMMARY

METHOD SW5030C/8260B  
 VOLATILE ORGANICS BY GC/MS

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=====
Client       : XYZ INC.                      Date Collected: 04/28/14
Project      : CLEAN WATER PROJECT           Date Received: 04/29/14
Batch No.    : YMNNN                         Date Extracted: 04/29/14 16:55
Sample ID    : XX-59XX001                   Date Analyzed: 04/29/14 16:55
Lab Samp ID  : MNNN-02                      Dilution Factor: 1
Lab File ID  : RDC534                       Matrix          : WATER
Ext Btch ID  : VO67D21                      % Moisture     : NA
Calib. Ref.  : RDC168                       Instrument ID   : 67
=====
  
```

PARAMETERS	RESULTS (ug/L)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)
BENZENE	0.11J	1.0	0.10	0.20
BROMODICHLOROMETHANE	ND	1.0	0.10	0.20
BROMOFORM	1.5	1.0	0.15	0.30
BROMOMETHANE	ND	1.0	0.16	0.30
CARBON TETRACHLORIDE	ND	1.0	0.10	0.20
CHLORO BENZENE	ND	1.0	0.10	0.20
CHLOROETHANE	ND	1.0	0.27	0.30
CHLOROFORM	ND	1.0	0.10	0.20
CHLOROMETHANE	ND	2.0	0.15	0.30
DIBROMOCHLOROMETHANE	0.20J	1.0	0.10	0.20
1,2-DICHLORO BENZENE	ND	1.0	0.10	0.20
1,3-DICHLORO BENZENE	ND	1.0	0.11	0.20
1,4-DICHLORO BENZENE	ND	1.0	0.10	0.20
DICHLORODIFLUOROMETHANE (FREON 12)	ND	1.0	0.15	0.30
1,1-DICHLOROETHANE	ND	1.0	0.10	0.20
1,2-DICHLOROETHANE	ND	1.0	0.10	0.20
1,1-DICHLOROETHENE	ND	1.0	0.10	0.20
1,2-DICHLOROETHENE (TOTAL)	0.30J	1.0	0.10	0.20
1,2-DICHLOROPROPANE	ND	1.0	0.10	0.20
TRANS-1,3-DICHLOROPROPENE	ND	1.0	0.11	0.20
CIS-1,3-DICHLOROPROPENE	ND	1.0	0.10	0.20
ETHYLBENZENE	ND	1.0	0.10	0.20
METHYLENE CHLORIDE	ND	5.0	0.25	0.50
1,1,2,2-TETRACHLOROETHANE	ND	1.0	0.11	0.20
TETRACHLOROETHENE	0.19J	1.0	0.15	0.20
TOLUENE	62	1.0	0.10	0.20
1,1,1-TRICHLOROETHANE	ND	1.0	0.10	0.20
1,1,2-TRICHLOROETHANE	ND	1.0	0.10	0.20
TRICHLOROFLUOROMETHANE (FREON 11)	ND	1.0	0.15	0.30
TRICHLOROETHENE	14	1.0	0.10	0.20
VINYL CHLORIDE	ND	2.0	0.12	0.20
XYLENES (TOTAL)	ND	1.0	0.10	0.50

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
1,2-DICHLOROETHANE-D4	8.44	10.00	84.4	70-120
4-BROMOFLUOROBENZENE	9.41	10.00	94.1	75-120
TOLUENE-D8	10.3	10.00	103	85-120
DIBROMOFLUOROMETHANE	9.95	10.00	99.5	85-115

## Figure 8: TYPICAL LCS/LCSD SUMMARY

### EMAX QUALITY CONTROL DATA LCS/LCD ANALYSIS

CLIENT: XYZ INC.  
 PROJECT: CLEAN WATER PROJECT  
 BATCH NO.: YMMNN  
 METHOD: SW5030C/8260B

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MATRIX: WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1W
LAB SAMP ID: VO67D21B VO67D21L VO67D21C
LAB FILE ID: RDC527 RDC524 RDC525
DATE EXTRACTED: 04/29/1413:15 04/29/1411:44 04/29/1412:15 DATE COLLECTED: NA
DATE ANALYZED: 04/29/1413:15 04/29/1411:44 04/29/1412:15 DATE RECEIVED: 04/29/14
PREP. BATCH: VO67D21 VO67D21 VO67D21
CALIB. REF: RDC168 RDC168 RDC168
  
```

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Benzene	ND	10.0	9.32	93	10.0	9.17	92	2	80-120	30
Bromodichloromethane	ND	10.0	9.65	97	10.0	9.37	94	3	75-120	30
Bromoform	ND	10.0	10.1	101	10.0	9.80	98	3	70-130	30
Bromomethane	ND	10.0	10.6	106	10.0	9.80	98	7	30-145	30
Carbon Tetrachloride	ND	10.0	8.35	83	10.0	8.20	82	2	65-140	30
Chlorobenzene	ND	10.0	9.78	98	10.0	9.68	97	1	80-120	30
Chloroethane	ND	10.0	11.0	110	10.0	10.1	101	8	60-135	30
Chloroform	ND	10.0	9.84	98	10.0	9.56	96	3	65-135	30
Chloromethane	ND	10.0	10.2	102	10.0	9.58	96	7	40-125	30
Dibromochloromethane	ND	10.0	9.64	96	10.0	9.49	95	2	60-135	30
1,2-Dichlorobenzene	ND	10.0	10.3	103	10.0	10.1	101	2	70-120	30
1,3-Dichlorobenzene	ND	10.0	10.1	101	10.0	9.91	99	2	75-125	30
1,4-Dichlorobenzene	ND	10.0	10.0	100	10.0	9.87	99	1	75-125	30
Dichlorodifluoromethane (Freon 12)	ND	10.0	9.93	99	10.0	9.39	94	6	30-155	30
1,1-Dichloroethane	ND	10.0	9.27	93	10.0	8.99	90	3	70-135	30
1,2-Dichloroethane	ND	10.0	7.97	80	10.0	7.88	79	1	70-130	30
1,1-Dichloroethene	ND	10.0	7.58	76	10.0	7.41	74	2	70-130	30
1,2-Dichloroethene (Total)	ND	20.0	17.1	85	20.0	16.6	83	3	70-125	30
1,2-Dichloropropane	ND	10.0	9.90	99	10.0	9.70	97	2	75-125	30
Trans-1,3-Dichloropropene	ND	10.0	8.39	84	10.0	8.49	85	1	55-140	30
cis-1,3-Dichloropropene	ND	10.0	9.23	92	10.0	8.93	89	3	70-130	30
Ethylbenzene	ND	10.0	9.84	98	10.0	9.67	97	2	75-125	30
Methylene Chloride	ND	10.0	8.28	83	10.0	8.40	84	1	55-140	30
1,1,2,2-Tetrachloroethane	ND	10.0	10.5	105	10.0	10.3	103	2	65-130	30
Tetrachloroethene	ND	10.0	9.31	93	10.0	9.20	92	1	45-150	30
Toluene	ND	10.0	9.57	96	10.0	9.47	95	1	75-120	30
1,1,1-Trichloroethane	ND	10.0	8.74	87	10.0	8.56	86	2	65-130	30
1,1,2-Trichloroethane	ND	10.0	10.3	103	10.0	10.3	103	0	75-125	30
Trichlorofluoromethane (Freon 11)	ND	10.0	10.8	108	10.0	9.93	99	9	60-145	30
Trichloroethene	ND	10.0	9.46	95	10.0	9.19	92	3	70-125	30
Vinyl Chloride	ND	10.0	10.3	103	10.0	9.61	96	7	50-145	30
Xylenes (Total)	ND	30.0	28.5	95	30.0	28.0	93	2	80-120	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT ( % )
1,2-Dichloroethane-d4	10.0	8.20	82	10.0	8.24	82	70-120
4-Bromofluorobenzene	10.0	9.69	97	10.0	9.70	97	75-120
Toluene-d8	10.0	10.4	104	10.0	10.4	104	85-120
Dibromofluoromethane	10.0	9.92	99	10.0	9.85	98	85-115

## Figure 9: TYPICAL MS/MSD SUMMARY

### EMAX QUALITY CONTROL DATA MS/MSD ANALYSIS

CLIENT: XYZ INC.  
PROJECT: CLEAN WATER PROJECT  
BATCH NO.: YMMNN  
METHOD: SW5030C/8260B

MATRIX: WATER % MOISTURE: NA  
DILUTION FACTOR: 1 1 1  
SAMPLE ID: XXX-59XX001 XXX-59XX001MS XXX-59XX001MSD  
LAB SAMP ID: MNNN-02 MNNN-02M MNNN-02S  
LAB FILE ID: RDC534 RDC535 RDC536  
DATE EXTRACTED: 04/29/1416:55 04/29/1417:26 04/29/1418:15 DATE COLLECTED: NA  
DATE ANALYZED: 04/29/1416:55 04/29/1417:26 04/29/1418:15 DATE RECEIVED: 04/29/14  
PREP. BATCH: VO67D21 VO67D21 VO67D21  
CALIB. REF: RDC168 RDC168 RDC168

#### ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Benzene	ND	10.0	9.32	93	10.0	9.17	92	2	80-120	30
Bromodichloromethane	ND	10.0	9.65	97	10.0	9.37	94	3	75-120	30
Bromoform	ND	10.0	10.1	101	10.0	9.80	98	3	70-130	30
Bromomethane	ND	10.0	10.6	106	10.0	9.80	98	7	30-145	30
Carbon Tetrachloride	ND	10.0	8.35	83	10.0	8.20	82	2	65-140	30
Chlorobenzene	ND	10.0	9.78	98	10.0	9.68	97	1	80-120	30
Chloroethane	ND	10.0	11.0	110	10.0	10.1	101	8	60-135	30
Chloroform	ND	10.0	9.84	98	10.0	9.56	96	3	65-135	30
Chloromethane	ND	10.0	10.2	102	10.0	9.58	96	7	40-125	30
Dibromochloromethane	ND	10.0	9.64	96	10.0	9.49	95	2	60-135	30
1,2-Dichlorobenzene	ND	10.0	10.3	103	10.0	10.1	101	2	70-120	30
1,3-Dichlorobenzene	ND	10.0	10.1	101	10.0	9.91	99	2	75-125	30
1,4-Dichlorobenzene	ND	10.0	10.0	100	10.0	9.87	99	1	75-125	30
Dichlorodifluoromethane (Freon 12)	ND	10.0	9.93	99	10.0	9.39	94	6	30-155	30
1,1-Dichloroethane	ND	10.0	9.27	93	10.0	8.99	90	3	70-135	30
1,2-Dichloroethane	ND	10.0	7.97	80	10.0	7.88	79	1	70-130	30
1,1-Dichloroethene	ND	10.0	7.58	76	10.0	7.41	74	2	70-130	30
1,2-Dichloroethene (Total)	ND	20.0	17.1	85	20.0	16.6	83	3	70-125	30
1,2-Dichloropropane	ND	10.0	9.90	99	10.0	9.70	97	2	75-125	30
Trans-1,3-Dichloropropene	ND	10.0	8.39	84	10.0	8.49	85	1	55-140	30
cis-1,3-Dichloropropene	ND	10.0	9.23	92	10.0	8.93	89	3	70-130	30
Ethylbenzene	ND	10.0	9.84	98	10.0	9.67	97	2	75-125	30
Methylene Chloride	ND	10.0	8.28	83	10.0	8.40	84	1	55-140	30
1,1,2,2-Tetrachloroethane	ND	10.0	10.5	105	10.0	10.3	103	2	65-130	30
Tetrachloroethene	ND	10.0	9.31	93	10.0	9.20	92	1	45-150	30
Toluene	ND	10.0	9.57	96	10.0	9.47	95	1	75-120	30
1,1,1-Trichloroethane	ND	10.0	8.74	87	10.0	8.56	86	2	65-130	30
1,1,2-Trichloroethane	ND	10.0	10.3	103	10.0	10.3	103	0	75-125	30
Trichlorofluoromethane (Freon 11)	ND	10.0	10.8	108	10.0	9.93	99	9	60-145	30
Trichloroethene	ND	10.0	9.46	95	10.0	9.19	92	3	70-125	30
Vinyl Chloride	ND	10.0	10.3	103	10.0	9.61	96	7	50-145	30
Xylenes (Total)	ND	30.0	28.5	95	30.0	28.0	93	2	80-120	30

SURROGATE PARAMETER	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	QC LIMIT ( % )
1,2-Dichloroethane-d4	10.0	8.20	82	10.0	8.24	82	70-120
4-Bromofluorobenzene	10.0	9.69	97	10.0	9.70	97	75-120
Toluene-d8	10.0	10.4	104	10.0	10.4	104	85-120
Dibromofluoromethane	10.0	9.92	99	10.0	9.85	98	85-115

**Figure 10:**

**TYPICAL CASE NARRATIVE**

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CASE NARRATIVE

Client : XYZ INC.

Project : CLEAN WATER PROJECT

SDG : YYDNNN

METHOD SW5030C/8260B  
VOLATILE ORGANICS BY GC/MS

A total of two (2) water samples were received on 04/29/14 for Volatile Organics by GC/MS analysis, Method 5030C/8260B in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods and Project QAPP Clean Water Project.

Holding Time

Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration

Instrument tune check was performed prior to calibration. Instrument mass ratios were within specification. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for VO67D21L/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at the frequency prescribed by the project. Percent recoveries and RPDs for MNNN-02M/S were within project QC limits.

Surrogate

Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter.

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURES**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2 <sup>nd</sup> Rvw
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in Table 5	Retune instrument and verify		
Multi point Initial Calibration(ICAL) minimum of 5 points	Initially; as needed	SPCCs : RF $\geq$ 0.1 for Bromoform, Chloromethane and 1,1-Dichloroethane RF $\geq$ 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane CCC: RSD $\leq$ 30% for the following analytes: Chloroform, 1,1-DCE, 1,2-DCP, Ethylbenzene, Toluene and Vinyl Chloride. 1.) if RRF is applied, then RSD $\leq$ 15% 2.) If 1st order is applied, then $r \geq$ 0.995 with min 5 pt ICAL 3.) If 2nd order is applied, then $r \geq$ 0.99 with min 6 pt ICAL	Check for outliers. Otherwise, optimize the instrument then repeat initial calibration.		
Initial calibration verification (ICV)	After initial calibration	All analytes within $\pm$ 20% of expected value except for the following compounds due to erratic chromatographic behavior: Bromomethane, Chloroethane, Chloromethane, Dichlorodifluoromethane but must be within $\pm$ 35% of expected value.	Verify second source standard. Prepare fresh standard and rerun ICV. If that fails, Optimize instrument and repeat ICAL.		
Evaluation of relative retention times (RRT)	Each sample	Within $\pm$ 0.06 RRT units	Correct the problem then reanalyze all samples analyzed since the last retention time check		
Continuing Calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs: Min. RF same as ICAL CCC : %Diff $\leq$ 20% (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct the problem then repeat initial calibration		
Internal Standard (IS)	All samples	Retention time $\pm$ 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning		
Method blank (MB)	One per preparation batch	No analytes detected $>$ $\frac{1}{2}$ LOQ	Rule out instrument contamination by re-analyzing the MB. If problem persist refer to PSR. In the absence of PSR, report NDs and results $>$ 10X of the MB concentration. Otherwise, cure contamination source, re-prep and re-analyze method blank and all associated samples.		
LCS	One LCS per preparation	Within project QC Limits	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD	One MS/MSD per every 20 project samples per matrix	Within project QC Limits	Check if sample was properly spiked. If indicative of matrix interference, discuss in case narrative, otherwise re-prep and re-analyze the sample		
Surrogate	Every Sample, MB, LCS, MS/MSD, DCC	Within project QC Limits	Correct the problem then re-analyze		
<b>Comments:</b> This QCP is applicable in the absence of the PSR Report values between LOD and LOQ. Refer to PSR for Flagging Criteria.				Reviewed by:	
				Date:	

## Appendix 2: DEMONSTRATION OF CAPABILITY for 25 ml

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

PARAMETER	RAY133	RAY134	RBV004	RBV005	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VOF5A10L	VOF5A10C	VOF5B01L	VOF5B01C							
Acetone	46.2	49.8	45.0	49.4	50	47.6	95	2.378	5	60 - 130	Passed
Acetonitrile	83.4	86.1	88.7	83.0	100	85.3	85	2.632	3	50 - 130	Passed
Acrolein	47.0	50.2	42.0	48.0	50	46.8	94	3.502	7	10 - 160	Passed
Acrylonitrile	50.0	52.1	45.0	51.3	50	49.6	99	3.188	6	60 - 150	Passed
Benzene	9.39	10.0	9.71	9.62	10	9.69	97	0.255	3	70 - 130	Passed
Bromobenzene	9.65	10.4	9.85	10.1	10	10.0	100	0.344	3	70 - 130	Passed
Bromochloromethane	9.08	9.78	9.18	9.48	10	9.4	94	0.315	3	70 - 130	Passed
Bromodichloromethane	9.58	10.1	9.81	10.0	10	9.9	99	0.239	2	70 - 130	Passed
Bromoform	9.70	10.3	9.00	10.0	10	9.76	98	0.568	6	60 - 130	Passed
Bromomethane	9.49	10.2	10.6	10.3	10	10.14	101	0.465	5	50 - 140	Passed
tert-Butyl alcohol	51.8	54.4	42.6	48.4	50	49.3	99	5.096	10	50 - 150	Passed
2-Butanone (MEK)	49.3	51.8	42.1	50.5	50	48.4	97	4.344	9	70 - 130	Passed
n-Butylbenzene	10.0	10.8	11.1	10.4	10	10.6	106	0.472	4	70 - 130	Passed
sec-Butylbenzene	9.94	10.8	10.8	10.5	10	10.5	105	0.397	4	70 - 130	Passed
tert-Butylbenzene	8.78	9.46	9.31	8.95	10	9.1	91	0.315	3	70 - 130	Passed
Carbon disulfide	9.24	10.2	8.45	8.74	10	9.15	92	0.755	8	50 - 130	Passed
Carbon tetrachloride	9.24	10.0	10.0	9.65	10	9.7	97	0.343	4	60 - 130	Passed
Chlorobenzene	9.08	9.68	9.39	9.22	10	9.3	93	0.257	3	70 - 130	Passed
2-Chloroethyl vinyl ether	9.55	9.91	8.16	9.38	10	9.3	93	0.760	8	10 - 160	Passed
Chloroethane	9.11	9.81	9.89	10.1	10	9.72	97	0.414	4	60 - 130	Passed
Chloroform	9.02	9.65	9.54	9.39	10	9.4	94	0.272	3	70 - 130	Passed
1-Chlorohexane	8.77	9.38	9.10	8.85	10	9.0	90	0.278	3	70 - 130	Passed
Chloromethane	9.12	9.52	9.39	9.53	10	9.39	94	0.191	2	50 - 130	Passed
2-Chlorotoluene	9.86	10.7	10.1	10.7	10	10.3	103	0.433	4	70 - 130	Passed
4-Chlorotoluene	9.70	10.4	11.1	9.9	10	10.3	103	0.611	6	70 - 130	Passed
Isopropyl ether (DIPE)	9.76	10.4	9.33	9.68	10	9.8	98	0.435	4	70 - 130	Passed
Dibromochloromethane	9.85	10.4	9.62	10.2	10	10.0	100	0.346	3	70 - 130	Passed
1,2-Dibromo-3-chloropropane	9.14	9.80	8.25	8.90	10	9.02	90	0.641	7	60 - 130	Passed
1,2-Dibromoethane	9.90	10.5	9.39	10.2	10	10.0	100	0.487	5	70 - 130	Passed
Dibromomethane	9.82	10.3	8.87	9.58	10	9.6	96	0.584	6	70 - 130	Passed
1,1-Dichloroethane	9.15	9.83	9.42	9.31	10	9.4	94	0.292	3	70 - 130	Passed
1,2-Dichloroethane	9.45	10.0	9.52	9.89	10	9.7	97	0.261	3	70 - 130	Passed
1,2-Dichlorobenzene	8.98	9.68	9.33	9.26	10	9.3	93	0.290	3	70 - 130	Passed
1,3-Dichlorobenzene	9.61	10.4	9.92	9.88	10	9.9	99	0.327	3	70 - 130	Passed
trans-1,4-Dichloro-2-Butene	10.6	11.5	9.58	10.8	10	10.6	106	0.812	8	50 - 140	Passed
1,4-Dichlorobenzene	9.82	10.6	10.1	10.1	10	10.2	102	0.340	3	70 - 130	Passed
Dichlorodifluoromethane	9.14	10.0	9.22	9.67	10	9.50	95	0.383	4	50 - 140	Passed
1,1-Dichloroethene	9.17	9.8	9.40	9.27	10	9.4	94	0.289	3	60 - 130	Passed
cis-1,2-Dichloroethene	9.40	10.1	9.71	9.60	10	9.7	97	0.285	3	70 - 130	Passed
trans-1,2-Dichloroethene	9.27	9.83	9.34	9.21	10	9.4	94	0.282	3	60 - 130	Passed
Dichlorofluoromethane	8.27	9.12	9.84	9.25	10	9.12	91	0.646	7	70 - 130	Passed
1,1-Dichloropropene	9.42	10.2	9.79	9.72	10	9.8	98	0.319	3	70 - 130	Passed
1,2-Dichloropropane	9.43	10.0	9.48	9.68	10	9.6	96	0.256	3	70 - 130	Passed
1,3-Dichloropropane	9.82	10.4	9.54	10.1	10	10.0	100	0.359	4	70 - 130	Passed

Unit: ug/L  
Sample Amount(ml): 25  
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12  
Analyzed by: D. Nguyen

**Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY for 25 ml**

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

Unit: ug/L  
Sample Amount(ml): 25  
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12  
Analyzed by: D. Nguyen

PARAMETER	RAY133	RAY134	RBV004	RBV005	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VOF5A10L	VOF5A10C	VOF5B01L	VOF5B01C							
2,2-Dichloropropane	9.07	10.4	10.2	9.65	10	9.8	98	0.598	6	60 - 140	Passed
cis-1,3-Dichloropropene	10.0	10.7	10.0	10.3	10	10.3	103	0.339	3	70 - 130	Passed
trans-1,3-Dichloropropene	9.30	9.75	9.06	9.42	10	9.4	94	0.288	3	70 - 130	Passed
tert-Butyl ethyl ether (ETB)	11.1	11.8	10.1	10.8	10	11.0	110	0.685	6	70 - 130	Passed
Ethyl Methacrylate	9.30	9.68	8.15	9.21	10	9.1	91	0.656	7	70 - 130	Passed
Ethylbenzene	9.47	10.1	10.0	9.68	10	9.8	98	0.300	3	70 - 130	Passed
2-Hexanone (MBK)	50.6	52.3	42.7	50.6	50	49.0	98	4.298	9	70 - 140	Passed
Hexachlorobutadiene	8.21	8.92	8.99	8.08	10	8.6	86	0.469	5	70 - 130	Passed
Iodomethane	9.16	10.0	9.31	9.21	10	9.43	94	0.417	4	50 - 150	Passed
Isopropylbenzene	10.4	11.2	11.0	10.8	10	10.9	109	0.380	3	70 - 130	Passed
p-Isopropyltoluene	10.1	11.0	11.0	10.4	10	10.6	106	0.423	4	70 - 130	Passed
Methylene Chloride	8.56	9.09	8.74	8.87	10	8.8	88	0.225	3	60 - 130	Passed
4-Methyl-2-pentanone (MIBK)	52.9	54.7	45.5	53.6	50	51.7	103	4.184	8	70 - 130	Passed
tert-Butyl methyl ether	10.1	10.7	8.99	10.1	10	10.0	100	0.723	7	60 - 130	Passed
Naphthalene	10.3	10.7	9.41	9.88	10	10.07	101	0.565	6	50 - 140	Passed
n-Propylbenzene	10.6	11.4	11.2	11.1	10	11.1	111	0.369	3	70 - 130	Passed
Styrene	9.63	10.4	10.0	9.8	10	10.0	100	0.311	3	70 - 130	Passed
tert-Amyl methyl ether (TAME)	11.3	12.0	10.2	11.1	10	11.1	111	0.747	7	60 - 140	Passed
1,1,1,2-Tetrachloroethane	9.32	9.91	9.59	9.54	10	9.6	96	0.246	3	70 - 130	Passed
1,1,1,2-Tetrachloroethane	9.92	10.6	9.52	10.5	10	10.1	101	0.501	5	60 - 130	Passed
Tetrachloroethene	9.08	9.77	9.50	9.01	10	9.3	93	0.356	4	60 - 130	Passed
Toluene	9.68	10.3	10.1	9.94	10	10.0	100	0.262	3	70 - 130	Passed
1,1,1-Trichloroethane	8.83	10.0	10.2	9.52	10	9.6	96	0.591	6	70 - 130	Passed
1,1,2-Trichloroethane	9.67	10.1	9.45	10.1	10	9.8	98	0.346	4	70 - 130	Passed
1,2,3-Trichlorobenzene	8.77	9.14	8.46	8.40	10	8.7	87	0.339	4	60 - 130	Passed
1,2,4-Trichlorobenzene	9.04	9.63	8.93	8.60	10	9.1	91	0.432	5	60 - 140	Passed
Trichloroethene	9.21	9.93	9.42	9.29	10	9.5	95	0.326	3	70 - 130	Passed
Trichlorofluoromethane	9.06	9.62	10.1	10.3	10	9.76	98	0.544	6	60 - 140	Passed
1,2,3-Trichloropropane	9.93	10.5	9.45	10.6	10	10.1	101	0.524	5	70 - 130	Passed
1,1,2-Trichloro-1,2,2-trifluoroethane	8.70	9.49	9.45	9.05	10	9.2	92	0.376	4	60 - 150	Passed
1,2,4-Trimethylbenzene	9.8	10.6	10.6	10.2	10	10.3	103	0.374	4	70 - 130	Passed
1,3,5-Trimethylbenzene	9.9	10.7	10.5	10.2	10	10.3	103	0.335	3	70 - 130	Passed
Vinyl Acetate	10.9	11.0	9.68	11.1	10	10.66	107	0.662	6	40 - 150	Passed
Vinyl Chloride	8.26	8.56	9.02	9.01	10	8.71	87	0.372	4	60 - 130	Passed
m-Xylene & p-xylene	19.2	20.6	20.4	19.6	20	19.9	100	0.633	3	60 - 140	Passed
o-Xylene	8.80	9.41	9.18	8.93	10	9.1	91	0.270	3	70 - 130	Passed
1,2-Dichloroethane-d4	10.0	10.0	9.9	10.3	10	10.05	101	0.200	2	70 - 130	Passed
Toluene-d8	10.3	10.3	10.4	10.3	10	10.32	103	0.046	0.5	70 - 130	Passed
4-Bromofluorobenzene	10.6	10.6	10.5	10.9	10	10.68	107	0.189	2	70 - 130	Passed
Dibromofluoromethane	10.0	9.93	10.1	10.2	10	10.06	101	0.106	1	70 - 130	Passed

### Appendix 3: DEMONSTRATION OF CAPABILITY for 5 ml

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

PARAMETER	RAN044	RAN045	RAN058	RAN059	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VSF4A07L	VSF4A07C	VSF4A08L	VSF4A08C							
Acetone	261	261	275	272	250	267	107	7.569	3	60 130	Passed
Acetonitrile	540	546	510	533	500	532	106	15.685	3	30 160	Passed
Acrolein	334	330	337	325	250	331	133	5.396	2	30 160	Passed
Acrylonitrile	269	270	278	271	250	272	109	4.119	2	70 130	Passed
Benzene	51.0	52.3	48.2	49.6	50	50.3	101	1.744	3	70 130	Passed
Bromobenzene	46.8	48.0	45.4	46.2	50	46.6	93	1.104	2	70 130	Passed
Bromochloromethane	51.7	52.2	50.7	51.2	50	51.5	103	0.611	1	70 130	Passed
Bromodichloromethane	49.5	50.8	48.4	49.2	50	49.5	99	1.022	2	70 130	Passed
Bromoform	47.8	48.6	45.6	47.5	50	47.4	95	1.282	3	70 130	Passed
Bromomethane	54.8	51.5	50.4	50.5	50	51.8	104	2.076	4	60 130	Passed
tert-Butyl alcohol	281	286	255	271	250	273	109	13.969	5	60 140	Passed
2-Butanone (MEK)	279	278	284	282	250	281	112	3.049	1	70 130	Passed
n-Butylbenzene	53.8	54.5	49.7	50.6	50	52.2	104	2.387	5	70 130	Passed
sec-Butylbenzene	52.0	52.5	47.9	49.4	50	50.5	101	2.181	4	70 130	Passed
tert-Butylbenzene	52.4	52.3	48.2	49.6	50	50.6	101	2.047	4	70 130	Passed
Carbon disulfide	48.2	46.9	44.2	46.0	50	46.4	93	1.669	4	60 130	Passed
Carbon tetrachloride	52.9	53.1	49.7	51.2	50	51.7	103	1.588	3	70 130	Passed
Chlorobenzene	49.3	50.2	47.1	47.9	50	48.6	97	1.402	3	70 130	Passed
2-Chloroethyl vinyl ether	50.1	49.7	36.4	38.4	50	43.7	87	7.258	17	50 150	Passed
Chloroethane	58.5	52.1	53.0	53.1	50	54.2	108	2.944	5	70 130	Passed
Chloroform	52.1	52.6	50.9	51.3	50	51.7	103	0.749	1	70 130	Passed
1-Chlorohexane	56.0	56.6	50.6	51.8	50	53.7	107	2.974	6	70 130	Passed
Chloromethane	53.8	48.8	48.5	50.0	50	50.3	101	2.456	5	60 130	Passed
2-Chlorotoluene	49.0	48.9	45.9	46.6	50	47.6	95	1.619	3	70 130	Passed
4-Chlorotoluene	51.9	53.0	49.5	49.8	50	51.1	102	1.675	3	70 130	Passed
2-Chloro-1,1,1-trifluoroethane	54.6	53.8	51.2	54.3	50	53.5	107	1.562	3	30 160	Passed
Chlorotrifluoroethylene	46.4	44.7	41.6	45.6	50	44.6	89	2.093	5	30 160	Passed
Dibromochloromethane	48.4	49.7	47.4	47.9	50	48.4	97	1.017	2	70 130	Passed
1,2-Dibromo-3-chloropropane	47.1	47.9	44.3	45.6	50	46.2	92	1.577	3	60 130	Passed
1,2-Dibromoethane	49.2	50.7	47.3	48.4	50	48.9	98	1.452	3	70 130	Passed
Dibromomethane	48.1	49.6	47.1	48.3	50	48.3	97	1.022	2	70 130	Passed
1,1-Dichloroethane	55.3	55.9	52.7	53.4	50	54.3	109	1.554	3	70 130	Passed
1,2-Dichloroethane	49.5	50.1	48.7	49.8	50	49.5	99	0.593	1	70 130	Passed
1,2-Dichlorobenzene	47.0	47.6	45.5	45.6	50	46.4	93	1.084	2	70 130	Passed
1,3-Dichlorobenzene	48.6	49.3	45.9	46.2	50	47.5	95	1.704	4	70 130	Passed
trans-1,4-Dichloro-2-Butene	51.0	50.7	48.7	50.0	50	50.1	100	1.012	2	70 130	Passed
1,4-Dichlorobenzene	49.0	49.7	46.6	46.7	50	48.0	96	1.599	3	70 130	Passed
Dichlorodifluoromethane	51.3	47.0	46.7	47.4	50	48.1	96	2.151	4	60 130	Passed
1,1-Dichloroethene	58.3	58.5	54.2	55.1	50	56.5	113	2.189	4	70 130	Passed
cis-1,2-Dichloroethene	53.3	53.5	50.9	51.2	50	52.2	104	1.358	3	70 130	Passed
trans-1,2-Dichloroethene	55.7	55.7	51.9	52.8	50	54.0	108	1.960	4	70 130	Passed
Dichlorofluoromethane	56.9	57.0	54.7	55.1	50	55.9	112	1.234	2	70 130	Passed
1,1-Dichloropropene	53.1	53.5	48.5	49.7	50	51.2	102	2.483	5	70 130	Passed
1,2-Dichloropropane	51.0	52.2	48.8	50.4	50	50.6	101	1.413	3	70 130	Passed

Unit: ug/L  
Sample Amount(ml): 5  
Sample Purge(ml): 5

Date Analyzed: 01/07/13 & 01/08/13  
Analyzed by: C. Mendoza

**Appendix 3 (cont.): DEMONSTRATION OF CAPABILITY for 5 ml**

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

PARAMETER	RAN044	RAN045	RAN058	RAN059	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	V5F4A07L	V5F4A07C	V5F4A08L	V5F4A08C							
1,3-Dichloropropane	49.4	50.8	47.8	49.3	50	49.3	99	1.251	3	70 130	Passed
2,2-Dichloropropane	57.2	57.1	54.0	54.5	50	55.7	111	1.706	3	70 140	Passed
cis-1,3-Dichloropropene	51.3	52.8	49.1	50.4	50	50.9	102	1.548	3	70 130	Passed
trans-1,3-Dichloropropene	51.2	52.4	49.6	50.5	50	50.9	102	1.191	2	70 130	Passed
tert-Butyl ethyl ether (ETB)	56.5	57.2	53.8	55.1	50	55.7	111	1.523	3	70 130	Passed
Ethyl Methacrylate	52.9	53.6	49.9	51.0	50	52	104	1.679	3	30 160	Passed
Ethylbenzene	51.6	52.9	48.5	49.6	50	50.7	101	1.946	4	70 130	Passed
2-Hexanone (MBK)	275	275	284	277	250	278.0	111	4.403	2	70 130	Passed
Hexachlorobutadiene	47.3	49.2	45.7	45.1	50	46.8	94	1.814	4	70 130	Passed
Iodomethane	40.7	39.4	36.9	39.0	50	39.0	78	1.595	4	60 130	Passed
Isopropyl ether (DIPE)	57.2	58.2	54.5	56.0	50	56.5	113	1.595	3	70 130	Passed
Isopropylbenzene	52.1	52.3	48.0	49.5	50	50.5	101	2.093	4	70 130	Passed
p-Isopropyltoluene	52.4	53.0	48.7	49.2	50	51	102	2.199	4	70 130	Passed
Methyl acetate	52.9	52.1	54.2	55.6	50	53.7	107	1.525	3	30 160	Passed
Methylene Chloride	50.4	51.6	49.6	50.2	50	50.5	101	0.856	2	70 130	Passed
4-Methyl-2-pentanone (MIBK)	278	279	283	276	250	278.7	111	2.713	1	70 130	Passed
tert-Butyl methyl ether	54.7	55.3	52.6	53.8	50	54.1	108	1.140	2	70 130	Passed
Naphthalene	43.7	44.3	40.8	39.8	50	42.2	84	2.180	5	60 140	Passed
n-Propylbenzene	51.9	52.3	47.9	49.3	50	50.4	101	2.098	4	70 130	Passed
Styrene	51.6	53.2	49.6	50.4	50	51.2	102	1.553	3	70 130	Passed
tert-Amyl methyl ether (TAME)	55.3	56.0	53.3	54.2	50	54.7	109	1.212	2	70 130	Passed
1,1,1,2-Tetrachloroethane	48.7	50.4	47.5	48.2	50	48.7	97	1.250	3	30 160	Passed
1,1,2,2-Tetrachloroethane	49.1	49.2	45.7	47.4	50	47.9	96	1.652	3	30 160	Passed
Tetrachloroethene	50.6	51.2	47.0	48.0	50	49.2	98	2.014	4	70 130	Passed
Toluene	51.3	52.4	48.6	49.7	50	50.5	101	1.718	3	70 130	Passed
1,1,1-Trichloroethane	55.0	55.2	52.6	52.9	50	53.9	108	1.405	3	30 160	Passed
1,1,2-Trichloroethane	48.3	50.1	46.3	48.0	50	48.2	96	1.553	3	30 160	Passed
1,2,3-Trichlorobenzene	48.0	49.6	46.0	44.8	50	47.1	94	2.140	5	70 130	Passed
1,2,4-Trichlorobenzene	50.8	51.5	46.7	45.5	50	48.6	97	2.965	6	70 130	Passed
Trichloroethene	50.7	51.5	47.7	48.3	50	49.5	99	1.834	4	70 130	Passed
Trichlorofluoromethane	61.7	55.1	54.8	55.3	50	56.7	113	3.322	6	70 140	Passed
1,2,3-Trichloropropane	47.8	47.7	45.6	47.4	50	47.1	94	1.036	2	70 130	Passed
1,1,2-Trichloro-1,2,2-trifluoroethane	57.4	56.8	52.9	53.6	50	55.2	110	2.264	4	70 - 130	Passed
1,2,4-Trimethylbenzene	51.5	52.0	48.2	49.2	50	50.2	100	1.810	4	70 130	Passed
1,3,5-Trimethylbenzene	51.7	52.3	48.0	49.2	50	50.3	101	2.016	4	70 130	Passed
Vinyl Acetate	65.3	59.2	58.1	58.5	50	60.3	121	3.366	6	50 140	Passed
Vinyl Chloride	59.8	52.9	54.5	54.5	50	55.4	111	3.040	5	70 140	Passed
m-Xylene & p-xylene	103	105	97.0	98.7	100	101.0	101	3.765	4	70 130	Passed
o-Xylene	52.3	53.5	49.4	50.7	50	51.5	103	1.786	3	70 130	Passed
Allyl Chloride	57.3	58.3	54.1	55.5	50	56.3	113	1.869	3	30 160	Passed

Unit: µg/L  
Date Analyzed: 01/07/13 & 01/08/13  
Sample Amount(ml): 5  
Analyzed by: C. Mendoza  
Sample Purge(ml): 5

## Appendix 4: DEMONSTRATION OF CAPABILITY for 5 g

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

PARAMETER	RCP061	RCP062	RCP066	RCP067	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VO02C04L	VO02C04C	VO02C06L	VO02C06C							
Acetone	259	264	288	281	250	273	109	14.006	5	40 - 140	Passed
Acetonitrile	480	506	528	546	500	515	103	28.361	6	50 - 150	Passed
Acrylonitrile	240	245	275	268	250	257	103	17.133	7	10 - 160	Passed
Benzene	50.1	49.3	49.9	49.6	50	49.7	99	0.364	1	70 - 130	Passed
Bromobenzene	49.3	47.4	48.9	49.1	50	48.7	97	0.838	2	70 - 130	Passed
Bromochloromethane	52.6	53.3	55.0	56.1	50	54.2	108	1.592	3	70 - 130	Passed
Bromodichloromethane	50.1	49.8	51.8	52.2	50	51.0	102	1.207	2	70 - 130	Passed
Bromoform	49.2	49.2	50.3	49.6	50	49.6	99	0.501	1	70 - 130	Passed
Bromomethane	46.4	46.6	45.4	46.7	50	46.3	93	0.585	1	60 - 130	Passed
tert-Butyl alcohol	254	266	274	285	250	270	108	13.169	5	40 - 150	Passed
2-Butanone (MEK)	246	251	271	260	250	257	103	11.327	4	60 - 140	Passed
n-Butylbenzene	53.2	51.3	52.5	53.9	50	52.7	105	1.105	2	70 - 130	Passed
sec-Butylbenzene	52.1	51.3	51.9	52.3	50	51.9	104	0.441	1	70 - 130	Passed
tert-Butylbenzene	48.4	48.0	48.7	49.4	50	48.6	97	0.572	1	70 - 130	Passed
Carbon disulfide	48.1	49.8	52.1	42.5	50	48.1	96	4.087	8	60 - 130	Passed
Carbon tetrachloride	52.5	51.2	52.2	51.2	50	51.8	104	0.666	1	70 - 130	Passed
Chlorobenzene	51.3	51.3	49.8	51.2	50	50.9	102	0.728	1	70 - 130	Passed
2-Chloroethyl vinyl ether	57.8	57.8	51.8	51.2	50	54.6	109	3.681	7	50 - 140	Passed
Chloroethane	55.0	56.1	52.3	53.3	50	54.2	108	1.696	3	70 - 140	Passed
Chloroform	55.4	55.5	53.0	54.3	50	54.5	109	1.171	2	70 - 130	Passed
1-Chlorohexane	53.3	51.9	51.8	52.2	50	52.3	105	0.678	1	70 - 130	Passed
Chloromethane	49.5	51.4	50.8	52.4	50	51.0	102	1.201	2	60 - 130	Passed
2-Chlorotoluene	44.7	48.4	45.5	48.0	50	46.6	93	1.814	4	70 - 130	Passed
4-Chlorotoluene	57.2	51.0	56.4	52.5	50	54.3	109	3.031	6	60 - 130	Passed
Dibromochloromethane	49.5	49.7	51.1	52.9	50	50.8	102	1.574	3	70 - 130	Passed
1,2-Dibromo-3-chloropropane	44.9	46.0	47.8	48.2	50	46.7	93	1.570	3	60 - 130	Passed
1,2-Dibromoethane	47.2	48.1	50.3	50.4	50	49.0	98	1.600	3	70 - 130	Passed
Dibromomethane	48.8	48.5	49.8	50.6	50	49.4	99	0.957	2	70 - 130	Passed
1,1-Dichloroethane	55.4	55.2	54.5	55.8	50	55.2	110	0.518	1	70 - 130	Passed
1,2-Dichloroethane	51.3	50.9	51.7	51.1	50	51.3	103	0.310	1	70 - 130	Passed
1,2-Dichlorobenzene	50.3	50.3	49.0	50.0	50	49.9	100	0.602	1	70 - 130	Passed
1,3-Dichlorobenzene	50.6	50.0	50.0	50.8	50	50.3	101	0.429	1	70 - 130	Passed
trans-1,4-Dichloro-2-Butene	48.8	47.7	53.8	52.2	50	50.6	101	2.848	6	60 - 140	Passed
1,4-Dichlorobenzene	51.7	51.0	49.8	50.8	50	50.8	102	0.775	2	70 - 130	Passed
Dichlorodifluoromethane	66.9	67.2	58.9	59.2	50	63.1	126	4.620	7	60 - 130	Passed
1,1-Dichloroethene	51.9	53.0	52.1	54.3	50	52.8	106	1.104	2	60 - 130	Passed
cis-1,2-Dichloroethene	49.5	49.2	53.0	54.8	50	51.6	103	2.725	5	70 - 130	Passed
trans-1,2-Dichloroethene	53.2	52.2	54.2	55.1	50	53.7	107	1.267	2	70 - 130	Passed
Dichlorofluoromethane	49.3	51.4	53.2	56.7	50	52.6	105	3.134	6	70 - 130	Passed
1,1-Dichloropropene	51.5	49.7	50.8	50.5	50	50.6	101	0.753	1	70 - 130	Passed
1,2-Dichloropropane	50.5	49.7	52.0	51.8	50	51.0	102	1.108	2	70 - 130	Passed
1,3-Dichloropropane	47.6	47.9	50.9	51.5	50	49.5	99	2.010	4	70 - 130	Passed
2,2-Dichloropropane	56.3	55.0	54.4	55.9	50	55.4	111	0.857	2	60 - 140	Passed
cis-1,3-Dichloropropene	51.7	51.3	52.3	52.4	50	52.0	104	0.517	1	70 - 130	Passed

Unit: µg/Kg

Date Analyzed: 03/20 & 03/21/12

Sample Amount(g): 5

Analyzed by: C. Mendoza

Sample Purge(ml): 5

**Appendix 4 (cont.): DEMONSTRATION OF CAPABILITY for 5 g**

DEMONSTRATION OF CAPABILITY  
METHOD: SW 8260

PARAMETER	RCP061	RCP062	RCP066	RCP067	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
	VO02C04L	VO02C04C	VO02C06L	VO02C06C							
trans-1,3-Dichloropropene	52.6	52.3	53.1	53.7	50	52.9	106	0.623	1	70 - 130	Passed
1,4-Dioxane	878	878	947	958	1000	915	92	43.314	5	50 - 150	Passed
tert-Butyl ethyl ether (ETBE)	54.0	53.9	54.2	55.2	50	54.3	109	0.617	1	70 - 130	Passed
Ethyl Methacrylate	49.5	49.8	51.2	51.4	50	50.5	101	0.964	2	70 - 130	Passed
Ethylbenzene	50.5	50.8	50.7	51.3	50	50.8	102	0.355	1	70 - 130	Passed
2-Hexanone (MBK)	241	247	270	256	250	253	101	12.291	5	20 - 160	Passed
Hexachlorobutadiene	52.6	48.4	49.6	49.2	50	49.9	100	1.873	4	70 - 130	Passed
Iodomethane	61.3	61.7	50.7	52.5	50	56.6	113	5.760	10	60 - 140	Passed
Isopropyl ether (DIPE)	53.2	53.2	54.7	55.5	50	54.2	108	1.156	2	70 - 130	Passed
Isopropylbenzene	55.9	53.8	51.7	50.4	50	52.9	106	2.417	5	70 - 130	Passed
p-Isopropyltoluene	53.6	52.7	51.5	51.5	50	52.3	105	1.040	2	70 - 130	Passed
Methylene Chloride	48.6	49.4	49.9	51.6	50	49.9	100	1.276	3	70 - 130	Passed
4-Methyl-2-pentanone (MIBK)	243	249	266	253	250	253	101	9.792	4	50 - 150	Passed
tert-Butyl methyl ether	51.2	51.8	52.3	53.4	50	52.2	104	0.957	2	70 - 130	Passed
Naphthalene	49.7	47.2	48.9	48.7	50	48.6	97	1.018	2	60 - 140	Passed
n-Propylbenzene	50.2	48.4	51.1	49.9	50	49.9	100	1.136	2	70 - 130	Passed
Styrene	52.6	53.1	51.7	53.0	50	52.6	105	0.637	1	70 - 130	Passed
tert-Amyl methyl ether (TAME)	51.9	52.9	52.4	53.9	50	52.8	106	0.850	2	70 - 130	Passed
1,1,1,2-Tetrachloroethane	50.7	51.8	50.8	52.0	50	51.3	103	0.670	1	70 - 130	Passed
1,1,2,2-Tetrachloroethane	48.3	48.6	50.5	49.7	50	49.3	99	1.013	2	70 - 130	Passed
Tetrachloroethene	52.5	50.5	50.0	50.2	50	50.8	102	1.184	2	70 - 130	Passed
Toluene	49.9	49.3	50.1	50.1	50	49.9	100	0.359	1	70 - 130	Passed
1,1,1-Trichloroethane	55.4	55.0	53.7	54.8	50	54.7	109	0.756	1	70 - 130	Passed
1,1,2-Trichloroethane	49.6	49.4	50.3	51.7	50	50.2	100	1.049	2	70 - 130	Passed
1,2,3-Trichlorobenzene	52.9	49.1	50.9	50.8	50	50.9	102	1.545	3	70 - 130	Passed
1,2,4-Trichlorobenzene	56.6	52.9	52.3	54.2	50	54.0	108	1.911	4	70 - 140	Passed
Trichloroethene	51.4	50.6	49.6	49.4	50	50.3	101	0.955	2	70 - 130	Passed
Trichlorofluoromethane	63.3	61.5	57.5	55.8	50	59.6	119	3.486	6	70 - 140	Passed
1,2,3-Trichloropropane	45.2	45.3	48.9	48.3	50	46.9	94	1.942	4	70 - 130	Passed
1,1,2-Trichloro-1,2,2-trifluoroethane	57.2	58.5	50.7	54.4	50	55.2	110	3.450	6	70 - 140	Passed
1,2,4-Trimethylbenzene	52.6	51.3	51.3	51.4	50	51.7	103	0.647	1	70 - 130	Passed
1,3,5-Trimethylbenzene	50.7	49.9	51.0	51.1	50	50.7	101	0.516	1	70 - 130	Passed
Vinyl Acetate	50.9	47.8	59.8	57.1	50	53.9	108	5.524	10	20 - 160	Passed
Vinyl Chloride	55.2	57.3	51.7	52.9	50	54.3	109	2.495	5	60 - 140	Passed
m-Xylene & p-xylene	102	101	101	104	100	102.0	102	1.343	1	70 - 130	Passed
o-Xylene	49.9	50.3	51.3	52.6	50	51.0	102	1.217	2	70 - 130	Passed

Unit: µg/Kg  
Sample Amount(g): 5  
Sample Purge(ml): 5

Date Analyzed: 03/20 & 03/21/12  
Analyzed by: C. Mendoza



8260FA:

ANALYTICAL RUN LOG



ANALYSIS LOG FOR VOLATILES

SOP  EMAX-8260 Rev.No. \_\_  EMAX-624 Rev.No. \_\_  EMAX-8260SIM Rev.No. \_\_  EMAX-TCP5IM Rev.No. \_\_  EMAX-M8260SIM Rev.No. \_\_  EMAX-8260C Rev. No. \_\_

Start Date:  5-mL Purge  10-mL Purge  25-mL Purge

Book # A06 -053

Sample Prep ID	Data File Name	Lab Sample ID	Sample Amount	DF	Matrix			Notes	Instrument No. 06			
					INITIAL CALIBRATION REFERENCE				STANDARDS			
					W	S			NAME	ID	Amount (µl)	Conc. (mg/L)
01					pH < 2	Cl <sub>2</sub> < 5ppm			DATE			
02								ICAL ID				
03									STANDARDS			
04												
05									DCC			
06									DCC			
07									DCC			
08									DCC			
09									BFB			
10									IS/SURR.			
11									ICV/LCS			
12									ICV/LCS			
13									ICV/LCS			
14									ICV/LCS			
15									Data File Folder			
16										LOT #		
17									pH strip			
18									Chlorine strip			
19									Methanol			
20									NaHSO <sub>4</sub>			
21									Reagent Water			
22									Sand			
23									Electronic Data Archival Location	Date		
24									HPCHEM_VOA/TO06			
25									Comments:			
26												
27												
28												
29									<input type="checkbox"/> Refer to sample weight log			
30									Analyzed By:			
									Date Disposed:	Disposed By:		

BATCH



## STANDARD OPERATING PROCEDURES

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

Prepared By: Souzan Grease *Souzan Grease* Date: 07-09-14

Approved By: Kenette Pimentel *K. Pimentel* Date: 07-09-14  
QA Manager

Approved By: Caspar Pang *C. Pang* Date: 07-09-14  
Laboratory Director

Control Number: 8270-06-

**1.0** SCOPE AND APPLICATION

- 1.1. This method is used to determine the concentration of semivolatife organic compounds extracted from many types of solid waste matrices, soils, air sampling media and water samples. Analytes that are listed in Table 9 were determined when this SOP was established. Additional analytes may be added upon completion of similar validation as the analytes that are listed in Table 8 & 9.
- 1.2. This SOP is an adaptation of method SW846 8270C.

**2.0** SUMMARY OF METHOD

- 2.1. Samples are extracted with methylene chloride. Extracts are concentrated and appropriate cleanup procedure is applied if necessary.
- 2.2. Internal standards are added to an aliquot of the final extract and are qualitatively and quantitatively analyzed by gas chromatography equipped with mass spectrometry (GC/MS).
- 2.3. **Interference**
  - 2.3.1. Solvents, reagents, glassware, and other sample processing devices are all possible sources of artifacts and/or interferences to sample analysis. Hence, quality control of chemicals and proper decontamination of re-usable glassware and proofing the analytical instrument to be free from contamination shall be observed.
  - 2.3.2. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Determine source of interference in the preparation and/or cleanup of the samples and Perform corrective action to eliminate the problem.
  - 2.3.3. Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, rinse the sample syringe with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.
  - 2.3.4. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

**3.0** DETECTION LIMITS

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**
  - 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**

SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

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3.1.2. Refer to Table 8 & 9 for established limits.

**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and requires dilution for proper quantitation.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.
- 4.3. The dynamic range established for this method are:

<u>Water (µg/L)</u>	<u>Soil (µg/kg)</u>
10 - 100 µg/L	330 - 3300 µg/kg

**5.0 SAMPLE HOLDING TIME & PRESERVATION****5.1. Sample Preservation**

- 5.1.1. Store water and soil samples at ≤ 6°C away from light without freezing.
- 5.1.2. Store all extracts at ≤ 6°C without freezing.

**5.2. Holding Time**

- 5.2.1. Extract water samples within 7 days from sampling date.
- 5.2.2. Extract soil samples within 14 days from sampling date.
- 5.2.3. Analyze all extracts within 40 days from extraction completion date.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-1311 TCLP for Organic and Inorganic Analytes
- 6.2. EMAX-3520 Extraction, Continuous Liquid/Liquid
- 6.3. EMAX-3540 Extraction, Soxhlet
- 6.4. EMAX-3546 Extraction, Microwave
- 6.5. EMAX-3550 Extraction, Pulse Sonication
- 6.6. EMAX-3580 Waste Dilution
- 6.7. EMAX-3640 Cleanup, GPC
- 6.8. EMAX-DM01 Data Flow and Review
- 6.9. EMAX-QA04 Detection Limit (DL)
- 6.10. EMAX-QA05 Training
- 6.11. EMAX-QA08 Corrective Action

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

- 6.12. EMAX-QC01 Quality Control for Chemicals
- 6.13. EMAX-QC02 Analytical Standard Preparation
- 6.14. EMAX-QC07 Glassware Cleaning
- 6.15. EMAX-SM01 Sample Management
- 6.16. EMAX-SM03 Waste Disposal
- 6.17. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all SDS for all chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS & REAGENTS****8.1. Instruments and Supplies**

Gas Chromatography	Agilent Technologies 7890A with split/splitless injection, Shimadzu GC-17A, or equivalent
Mass Spectrometer	Agilent Technologies 5975C MSD or Shimadzu GCMS – GP 5000 capable of scanning from 1.6 to 1050 amu every 1 second using 70 volts electrode energy in the electron impact ionization mode or equivalent
GC/MS Interface	Capillary-direct into the mass spectrometer source or equivalent
Chromatographic Column	ZB-5MS (20 m x 0.18 mm x 0.32 µm) or equivalent
Data System	MS-ChemStation with Enviroquant software or equivalent
GC Autosampler	Agilent Technologies 7683B series injector or Shimadzu AOC-20i capable of direct injection of 1 µL and 10 µL of extract.
Gases	Ultra high purity helium
Syringes	10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL and 1000 µL syringe Hamilton 202N or equivalent
Vials	Autosampler vials with teflon lined septa

**8.2. Chemicals and Reagents**

Methylene chloride, pesticides grade
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## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

Methanol, high purity
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**9.0 STANDARDS****9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX-SM04, respectively.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

**9.2. Stock Standard**

- 9.2.1. Purchase stock standards as certified solutions from a reputable vendor (refer to Table 1 for the listing of all certified solutions). Standards are expected to be at least 96% purity. Read vendor's note if correction has been applied to certified values, otherwise corrections must be applied.
- 9.2.2. Transfer the stock standard solutions into 2 mL amber vial with Teflon lined screw caps and store at -10°C to -20°C.

**9.3. Intermediate Standard**

- 9.3.1. Using stock standard solutions, prepare the intermediate standard in Methylene Chloride according to Table 1.

**9.4. Internal Standard**

- 9.4.1. The internal standard shall include 1,4-Dichlorobenzene-d<sub>4</sub>, Naphthalene-d<sub>8</sub>, Acenaphthalene-d<sub>10</sub>, Phenanthrene-d<sub>10</sub>, Chrysene-d<sub>12</sub> and Perylene-d<sub>12</sub> in methylene chloride solution.
- 9.4.2. Purchase internal standard solutions as certified solution from a reputable vendor at 4,000 µg/mL.
- 9.4.3. Prepare a 10 mL of 2,000 µg/mL of working internal standard from 4,000 µg/mL (refer to Table 4). Transfer the solution in a properly labeled 10 mL amber vial and store in -10°C to -20°C.

**9.5. GC/MS Tuning**

- 9.5.1. The tuning standard shall include decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol, and Benzidine.
- 9.5.2. Purchase tuning standard solution as certified standard at 1000 µg/mL.
- 9.5.3. Prepare a 500 µL of 50 µg/mL of working standard tuning solution (refer to Table 5). Transfer the solution in a 1 mL amber vial and store in -10°C to -20°C.

**9.6. Surrogate Standard**

- 9.6.1. Purchase surrogate stock standards as certified standard.
  - 9.6.1.1. The acid surrogate mixture includes Phenol-d<sub>5</sub>, 2-Fluorophenol, and 2,4,6-Tribromophenol at 150 µg/mL.

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**

SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

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9.6.1.2. The basic neutral surrogate mixture includes Nitrobenzene-d<sub>5</sub>, 2-Fluorobiphenyl, Terphenyl-d<sub>14</sub>, and 1,2-Dichlorobenzene-d<sub>4</sub> at 50 µg/mL.

9.6.2. For typical extraction [soil: 30 g – 2 mL or water: 1000 mL – 2 mL], add 0.4 mL of surrogate spiking standard to the sample prior to extraction. Spike volume may be adjusted to normalize with the final extract and yield the same concentration.

**9.7. Calibration Standard**

9.7.1. Prepare working standard solutions for initial calibration and daily calibration (refer to Tables 2A-2C). Transfer the solutions in a 1 mL amber vial and store them at ≤ 6°C without freezing.

**9.8. ICAL Verification Standard (Second Source Verification) (ICV)**

9.8.1. Purchase a certified ICV standard from a different vendor. The ICV standard contains the same list of compounds as the stock standard (refer to Table 1B for the standard mix and the corresponding vendors).

9.8.2. Prepare a 500 µL of 25 µg/mL check standard solution (refer to Table 3A-3C). Transfer the solution in a properly labeled 1 mL amber vial and store at ≤ 6°C without freezing.

**9.9. LCS/MS Spiking Standards**

9.9.1. Purchase spiking standards as certified solutions from a different source.

9.9.2. Refer to specific extraction SOP for spiking amount used for LCS/LCSD/MS/MSD, unless otherwise specified by project.

**10.0 PROCEDURES****10.1. Sample Preparation**

10.1.1. For aqueous samples (including TCLP leachates), refer to EMAX-3520 or EMAX-1311. Record in the extraction log if presence of residual chlorine is observed.

10.1.2. For solid samples, refer to EMAX-3550, EMAX-3540 or EMAX-3546.

10.1.3. For waste samples, refer to EMAX-3580.

10.1.4. After extraction, examine the color and consistency of the extract. If the extract appears to be opaque and/or viscous, it is advisable to perform extract cleanup preferably GPC. Refer to EMAX-3640.

**10.2. Instrument Parameters**

10.2.1. Set the instrument parameters as suggested in Table 6. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and display current condition on the instrument for easy access when performing daily instrument routine check.

10.2.3. In the event that instrument parameters necessitate a change, replace the instrument parameter printout with the new parameter setup. Archive the previous instrument parameters in the instrument maintenance log.

10.2.4. Set injection volume to 1 µL to 2 µL.

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14**10.3. Calibration**

10.3.1. Set GC/MS operating condition as described in Section 10.2.

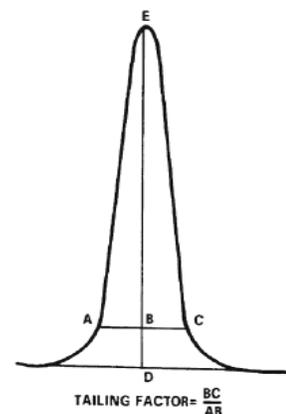
10.3.2. Perform Tune Check

10.3.2.1. Analyze a solution containing 50 µg/mL of tuning standard working solution, DFTTP, DDT, benzidine and pentachlorophenol.

10.3.2.2. A valid Tune Check expires after 12 hours. Evaluate the tune check by averaging three scans (the peak apex scan and the scan immediately preceding and the scan immediately following the apex). Apply a background subtraction using a single scan no more than 20 scans prior to the elution of DFTPP. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP. Use the DFTPP mass intensity criteria in the manufacturer's instructions as primary tuning acceptance criteria, otherwise refer to Table 7 for acceptance criteria. A valid tune expires after 12 hours.

10.3.2.3. Evaluate column performance and injection port inertness using the data acquisition software.

- Degradation of DDT to DDE and DDD must be less than 20% based on area obtained from the total ion chromatogram.
- Benzidine and Pentachlorophenol must be present at their normal responses. Evaluate the tailing factor of benzidine and pentachlorophenol. Benzidine tailing  $\leq 3$  and pentachlorophenol  $\leq 5$ . Refer to the attached figure for peak evaluation.



Example calculation: Peak Height = DE = 100 mm  
 10% Peak Height = BD = 10 mm  
 Peak Width at 10% Peak Height = AC = 23 mm  
 AB = 11 mm  
 BC = 12 mm  
 Therefore: Tailing Factor =  $\frac{12}{11} \approx 1.1$

10.3.2.4. If tune check is non-compliant, refer to Section 12 for corrective action.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Optimize the instrument condition prior to ICAL.

- Ensure that instrument parameters are set up properly
- Ensure that there is no evidence of leak
- Ensure that instrument maintenance is performed on schedule

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

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- Ensure that instrument tune check and column performance is not indicative that it is at the threshold of failing the acceptance criteria.
- 10.3.3.3. Analyze a multi-point initial calibration curve as suggested in Figure 3 after a valid tune check.
- 10.3.3.4. Base quantitation of identified compounds on the integrated abundance from the EICP of the assigned primary characteristic ion (refer to Tables 8 & 9). For optimum output, assign internal standard to each compound based on the nearest retention time or as suggested on tables 8 and 9.
- 10.3.3.5. **Evaluate the ICAL Acceptance**
- 10.3.3.5.1. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:
- Check the established retention time window
  - Check the relative intensity of major ions
  - Adjust accordingly if necessary.
- 10.3.3.5.2. Evaluate retention time of each analyte with respect to the nearest internal standard. The relative retention time (RRT) of each analyte should agree within  $\pm 0.06$  RRT units.
- 10.3.3.5.3. At a minimum, evaluate System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) as specified in Appendix 1. The list of SPCCs and CCCs are provided in Tables 10 and 11.
- 10.3.3.5.4. Check RSD and Correlation Coefficient. If more than 10% of the compounds included with the initial calibration exceed the 15% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform necessary instrument maintenance and repeat calibration. Refer to 10.3.3.2., Section 12 for corrective action.
- 10.3.3.6. **Application of ICAL Curve for Quantitation**
- 10.3.3.6.1. Generate a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRFm), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq.-10.5.1.1, Eq.-10.5.1.2, Eq.-10.5.1.6 and Eq.-10.5.1.7 respectively.
- 10.3.3.6.2. If RSD is  $\leq 15\%$  average response factor may be applied.
- 10.3.3.6.3. Apply Inverse Weighting Factor ( $1/y$  or  $1/y^2$ ;  $y$  being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 15\%$  and correlation coefficient of  $\geq 0.995$ .
- 10.3.3.6.4. Apply linear least squares regression if past experience or priori knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 15\%$  and correlation of determination (COD)  $\geq 0.99$ .

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

10.3.3.6.5. It may be appropriate to force the regression through zero for specific analytes.<sup>1</sup> When exercising this option [as included in the data acquisition software], make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Analyze ICV to verify the concentration of the ICAL standards (Refer to Section 9.8).

10.3.4.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of  $\pm 30$  seconds indicate instrument malfunction. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (- 50% to + 100%).

10.3.4.5. Refer to Appendix 1 for ICV acceptance criteria and/or corrective action.

10.3.4.6. When non-compliant refer to Section 12 for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC to check the validity of the ICAL (refer to 9.7).

10.3.5.2. Check for completeness of analytes as described in Section 10.4.3.

10.3.5.3. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.5.4. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of  $\pm 30$  seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.5.5. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.6. Establish RRF of each analyte, calculate %D (Eq.-10.5.2.1) against the ICAL.

10.3.5.7. Refer to Appendix 1 for DCC acceptance criteria and/or corrective action,

10.3.5.8. When non-compliant refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Extract Preparation

10.4.1.1. Allow extracts to equilibrate with room temperature.

<sup>1</sup> SW846 Method 8000B, Section 7.5.3

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14

10.4.1.2. Measure 300 µL of extract, transfer into an autosampler vial.

10.4.1.3. Add 6 µL of 2000 µg/mL of internal standard (refer to 9.4).

10.4.1.4. Seal the vial with Teflon-lined septa cap.

10.4.2. Analytical Sequence

10.4.2.1. Analyze instrument blank.

10.4.2.2. Analyze DFTPP and evaluate tuning.

10.4.2.3. Analyze DCC and check ICAL validity.

10.4.2.4. Analyze Method Blank.

10.4.2.5. Analyze Lab Control Sample and Lab Control Sample Duplicate (if requested).

10.4.2.6. Analyze samples to a maximum number of 12-hours from time of DFTPP injection.

10.4.2.7. Analyze a pair of matrix spikes (MS/MSD) for every 20 samples of the same matrix.

10.4.2.8. Record analytical sequence in the analysis log.

10.4.3. Sample Result Evaluation

10.4.3.1. Check the QC criteria as soon as the data is available.

- ✓ Check method blank. If result is non-compliant and analyte in question is not detected in any sample or contamination is < 10X of the sample concentration, results maybe reportable. Verify with the PM if results can be reported.
- ✓ Compare the retention times of each Internal Standards (IS) to the ICAL mid point (must be ± 30 seconds).
- ✓ Compare the area of each IS acquired against the mid point of the ICAL. The Extracted ION Current Profile (EICP) must be within a factor of two (-50 to +100%).
- ✓ Check concentration of target analytes if calibration range is exceeded.
- ✓ Check surrogate recoveries against project specific requirements (PSR). In the absence of PSR, default to Appendix 1 QC limits.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. Note observations on the analysis log. When result arise to questionable result, e.g. inconsistency from the first analysis, consult the Supervisor for further action.

10.4.3.2. **Qualitative Identification**

- The intensities of the characteristic ions must maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.

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- 
- Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. For manual integration refer to EMAX-DM01.
  - Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomers peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.<sup>2</sup>

10.4.3.2.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative<sup>3</sup> (TIC). Execute LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library. Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting following the guidelines below.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within + 20%.

Example: for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.

- Molecular ions present in reference spectrum but not present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting analytes.
- Ions present in the reference spectrum but not present in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of the background contamination or co-eluting analytes. Data system library reduction programs can sometimes create these discrepancies.

10.4.3.2.2. **Reporting TICs**

- If the library search produces a match at or above 85%, report the analyte.
- If the library search produces more than one analyte at or above 85%, report the first analyte (highest).

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<sup>2</sup> SW846 Method 8270C, Section 7.6.1.4

<sup>3</sup> Library search is performed only when indicated in the PSR.

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- If the library search produces no matches at or above 85%, the compound should be reported as unknown.

**10.4.3.3. Quantitation**

- 10.4.3.3.1. Apply the appropriate quantitation method (Section 10.3.3.6). Calculate the concentration of any positively identified target analyte using Eq.-10.5.3. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

**10.4.3.4. Manual Integration**

- 10.4.3.4.1. Refer to EMAX-DM01, manual integration section.

**10.4.3.5. Dealing with Carryover**

- 10.4.3.5.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- 10.4.3.5.2. If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- 10.4.3.5.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.
- 10.4.3.5.4. To clean-up the system, run a blank sample. If improved result is noted repeat this process until no evidence of contamination is observed. Otherwise inform the Supervisor for further instruction.

**10.5. Calculations****10.5.1. Initial Calibration****10.5.1.1. Calculate for Relative Response Factor (RRF)**

$$RRF = \frac{(AX)(CIS)}{(AIS)(CX)} \quad \text{Eq.-10.5.1.1}$$

where:

- AX - Area of characteristic ion for the compound being measured
- AIS - Area of characteristic ion for the specific internal standard
- CX - Concentration of the compound being measured
- CIS - Concentration of the specific internal standard

**10.5.1.2. Calculate for Average Relative Response Factor (RRF<sub>m</sub>)**

$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq-10.5.1.2}$$

where:

- RRF<sub>m</sub> - average response factor

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**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-14 $\sum RRF$  - summation of response factors $n$  - number of measurements**10.5.1.3. Calculate for Least Square Linear Regression**

$$y = ax + b \quad \text{Eq.-10.5.1.3}$$

where:

 $y$  - Response Ratio (AX/AIS) $x$  - Amount Ratio (CX/CIS) $a$  - x1 = slope of the line

$$a = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

where:

 $\bar{x}$  - Average of amount ratios $\bar{y}$  - Average of response ratios $b$  - x0 = intercept of the line

$$b = \bar{y} - a * \bar{x}$$

**10.5.1.4. Calculate for Inverse Weighting Factor**

$$y = ax + b \quad \text{Eq.-10.5.1.4}$$

where:

 $y$  - Response Ratio (AX/AIS) $x$  - Amount Ratio (CX/CIS) $a$  - x1 = slope of the line

$$a = \frac{\sum [(x - x_a)(y - y_a)]}{\sum (x - x_a)^2}$$

where:

$$x_a = \sum [x(1/x)] / \sum (1/x)$$

$$y_a = \sum [y(1/x)] / \sum (1/x) \text{ or}$$

$$x_a = \sum [x(1/x^2)] / \sum (1/x^2)$$

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$$y_a = \frac{\sum[y(1/x^2)]}{\sum(1/x^2)}$$

b = x0 = intercept of the line

$$b = y_a - a * x_a$$

10.5.1.5. **Calculate Inverse Quadratic**

$$y = ax^2 + bx + c$$

Eq.-10.5.1.5

where:

y - Resp\_Ratio = **x0 + x1** \* Amt\_Ratio + **x2** \* (Amt\_Ratio)<sup>2</sup>

x - Amt\_Ratio

c - **x0 = Det 0/Det b**b - **x1 = Det 1/Det b**a - **x2 = Det 2/Det b**

$$W_i = \frac{\frac{1}{X_i}}{\sum_{i=1}^n \frac{1}{X_i}}$$

where:

X<sub>i</sub> - amount ratio = Conc of Std/Conc of ISY<sub>i</sub> - response ratio = Resp of Std/Resp of IS**Wi** - 1/X)/SUM(1/X)**<X>** - SUM(W<sub>i</sub>\*X<sub>i</sub>)**<Y>** - SUM(W<sub>i</sub>\*Y<sub>i</sub>)**<XX>** - SUM(W<sub>i</sub>\*(X<sub>i</sub>)<sup>2</sup>)**<XXX>** - SUM(W<sub>i</sub>\*(X<sub>i</sub>)<sup>3</sup>)**<XXXX>** - SUM(W<sub>i</sub>\*(X<sub>i</sub>)<sup>4</sup>)**<YY>** - SUM(W<sub>i</sub>\*(Y<sub>i</sub>)<sup>2</sup>)**<XY>** - SUM(W<sub>i</sub>\*X<sub>i</sub>\*Y<sub>i</sub>)**<XXY>** - SUM(W<sub>i</sub>\*(X<sub>i</sub>)<sup>2</sup>\*Y<sub>i</sub>)**<Yd2>** - SUM((Y<sub>i</sub>-<Y>)<sup>2</sup>\*W<sub>i</sub>)**Ye** - x0+x1\*X<sub>i</sub>+x2\*X<sub>i</sub><sup>2</sup>-<Y>**<Ye2>** - SUM(Ye<sup>2</sup>\*W<sub>i</sub>)

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Det b	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

Det 0	1	<X>	<XX>
	<X>	<XX>	<XXX>
	<Y>	<XY>	<XXY>

Det 1	1	<X>	<XX>
	<Y>	<XY>	<XXY>
	<XX>	<XXX>	<XXXX>

Det 2	<Y>	<XY>	<XXY>
	<X>	<XX>	<XXX>
	<XX>	<XXX>	<XXXX>

$$r^2 = \frac{\langle Y_e^2 \rangle}{\langle Y_d^2 \rangle}$$

$$ccf^2 = (r^2)^{1/2}$$

## 10.5.1.6. Calculate for Standard Deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}} \quad \text{Eq.-10.5.1.6}$$

where:

 $X_i$  - result at  $i^{\text{th}}$  measurement $\bar{x}$  - mean $n$  - number of measurements

## 10.5.1.7. Calculate for % relative standard deviation (%RSD)

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq.-10.5.1.7}$$

where:

 $SD$  - standard deviation $RRF_m$  - average response factor

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**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-1410.5.1.8. **Calculate the Relative Retention Time (RRT)**

$$RRT = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the Internal Standard}} \quad \text{Eq.-10.5.1.8}$$

10.5.2. Calibration Check/Continuing Calibration10.5.2.1. **Calculate Percent Difference (%D) when RRF<sub>m</sub> is used for quantitation**

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq.-10.5.2.1}$$

where:

RRF<sub>c</sub> - response factor from continuing calibration standardRRF<sub>m</sub> - average response factor10.5.2.2. **Calculate Percent Deviation (%D<sub>t</sub>) when applied calculation is other than ARF**

$$\%D_t = \frac{|T_t - T_f|}{T_t} * 100\% \quad \text{Eq.-10.5.2.2}$$

where:

T<sub>t</sub> - true value of standard in µg/LT<sub>f</sub> - found value of standard in µg/L

10.5.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.5.3.1. **Water Samples**

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \quad \text{Eq.-10.5.3.1}$$

where:

A<sub>x</sub> - area of characteristic ion for the compound to be measuredI<sub>s</sub> - amount of internal standard addedV<sub>e</sub> - extract final volume from sample extraction, usually 1 mLDF - dilution factor =  $\frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$ A<sub>is</sub> - area of characteristic ion for the internal standardRRF<sub>m</sub> - average response factorV<sub>i</sub> - volume of extract injected in µL, usually 1 µLV<sub>t</sub> - volume of water extracted in mL, usually 1000 mL

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**SEMIVOLATILE ORGANICS BY GC/MS**SOP No.: EMAX-8270 Revision No. 6 Date: 10-Jul-1410.5.3.2. **Soil/Sediment Samples (Dry weight basis)**

$$\text{Concentration}(\mu\text{g} / \text{Kg}) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \quad \text{Eq.-10.5.3.2}$$

where:

- $A_x$  - area of characteristic ion for the compound to be measured  
 $I_s$  - amount of internal standard injected in ng  
 $V_e$  - volume of extract in mL, usually 1 mL<sup>4</sup>  
 $DF$  - dilution factor =  $\frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$   
 $A_{is}$  - area of characteristic ion for the internal standard  
 $RRF_m$  - average response factor  
 $V_i$  - volume of extract injected in  $\mu\text{L}$ , usually 1  $\mu\text{L}$   
 $W_s$  - wet soil weight in kg  
 $DW$  - % solid =  $\frac{100 - \% \text{moisture}}{100}$

10.5.3.3. **Calculation for results subjected to cleanup shall have the following equations:**

$$\text{Concentration}(\mu\text{g} / \text{Kg}) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \cdot \frac{V_{bg}}{V_{ig}} \quad \text{Eq.-10.5.3.3.1}$$

$$\text{Concentration}(\mu\text{g} / \text{L}) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \cdot \frac{V_{bg}}{V_{ig}} \quad \text{Eq.-10.5.3.3.2}$$

where:

- $A_x$  - area of characteristic ion for the compound to be measured  
 $I_s$  - amount of internal standard injection in ng  
 $V_e$  - volume of extract in mL  
 $DF$  - dilution factor =  $\frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$   
 $A_{is}$  - area of characteristic ion for the compound to be measured  
 $RRF_m$  - average response factor  
 $V_i$  - volume of extract injected in  $\mu\text{L}$ , usually 1  $\mu\text{L}$   
 $W_s$  - wet soil weight in Kg

<sup>4</sup> For extracts subjected to GPC  $V_i=0.5\text{-mL}$

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$$DW - \%solid = \frac{100 - \%moisture}{100}$$

$V_t$  - volume of water extracted in mL

$V_{bg}$  - total volume of extract before GPC clean-up in mL

$V_{ig}$  - injected volume of extracts to GPC in mL

10.5.4. Base all sample result calculations on the ICAL curve, e.g., area ratio of  $A_x/A_{is}$  versus concentration using inverse weighting factor fitted to the initial calibration is also used for determination of sample concentration.

10.5.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.5.5.1. The area "Ax" and "Ais" are derived from total ion chromatogram. "Ais" refers to the closest internal standard (IS) free of interference.

10.5.5.2. RRF of the TIC is 1.

10.5.6. Accuracy and Precision

10.5.6.1. **Percent Recovery**

$$\% Recovery = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq.-10.5.6.1}$$

where:

$C_f$  - concentration found

$C$  - concentration of sample (use 0 for LCS)

$C_s$  - concentration of spike

10.5.6.2. **Relative Percent Difference (%RPD)**

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.6.2}$$

where:

$C_1$  - Measured concentration of the first sample aliquot

$C_2$  - Measured concentration of the second sample aliquot

10.5.7. DDT Degradation

$$\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}} (100) \quad \text{Eq.-10.5.7}$$

where:

$\%B$  - percent breakdown

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*ADDD* - area of DDD*ADDE* - area of DDE*ADDT* - area of DDT**10.6. Data Reduction**

- 10.6.1. Make a copy of the analytical run log and sample preparation log.
- 10.6.2. Highlight the data to be reported.
- 10.6.3. Print a copy of the raw data and the QC report.
- 10.6.4. Check that all positively identified analytes are within the calibration range.
- 10.6.5. Collate the reportable data separating the QC results from the sample results.
- 10.6.6. Keep all other data generated with the analytical folder marked with "For record only".

**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WDB1C.exe
- 10.7.2. Generate the sample results using F1NV3C.exe or F1N3C4.exe
- 10.7.3. Generate the QC summary using QCV3CN.exe or QCV3CN4.exe
- 10.7.4. Generate the Instrument Performance Check (ICAL and DCC) using F5SVTEST.exe.
- 10.7.5. Generate the IS and RT Summary using F8SV.exe.
- 10.7.6. Generate Lab Chronicle using LABCHRN1.exe
- 10.7.7. Generate the Case Narrative using CN1.exe
- 10.7.8. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
  - 10.7.8.1. Case Narrative
  - 10.7.8.2. Lab Chronicle
  - 10.7.8.3. Sample Results
  - 10.7.8.4. Method Blank Results
  - 10.7.8.5. LCS/LCSD Summary
  - 10.7.8.6. MS/MSD Summary
  - 10.7.8.7. Instrument Performance Check (ICAL)
  - 10.7.8.8. ICAL Summary
  - 10.7.8.9. ICV Summary
  - 10.7.8.10. Instrument Performance Check (DCC)
  - 10.7.8.11. IS and RT Summary
  - 10.7.8.12. DCC Summary

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10.7.8.13. Analytical Run Log

10.7.8.14. Sample Preparation Log

10.7.8.15. Non-Conformance Report (if any)

**10.8. Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.1.1. If any of the checkpoints below indicates a problem, re-analysis is required.

- ✓ Check internal standard area. They should be within -50 to +100% of the ICAL midpoint to be acceptable, otherwise follow PSR.
- ✓ Check retention time of each IS to the ICAL midpoint. They should be within  $\pm$  30 seconds to be acceptable, otherwise follow PSR.
- ✓ Check surrogate recoveries against Project Specific requirements (PSR). In the absence of PSR, default to in-house QC limits.
- ✓ Check concentrations of target analytes if calibration range is exceeded.

10.8.1.2. Review the attached log that they are properly filled.

10.8.1.3. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.4. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.2. Submit the analytical folder for secondary review.

**10.9. Preventive Maintenance**

10.9.1. Perform instrument routine preventive maintenance and record on instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.2. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Vacuum System Verification	Verify pressure. Perform system tune check	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check instrument performance	Daily prior to analysis

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	(e.g., Daily calibration check, instrument blank, DDT/Endrin breakdown).	
Source Cleaning	Remove and clean the Mass Spec ion source	Every 6 months or as necessary
Vacuum System Maintenance	Inspect vacuum pumps, and replace mechanical/diffusion pump oil	Every 6 months
Documentation	Record maintenance in instrument service logs	Daily prior to analysis to include services done by third party.

**11.0 QUALITY CONTROL****11.1. Analytical Batch QC**

- 11.1.1. Perform tune check to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to calibration and check for any contamination.
- 11.1.2. Perform Initial Calibration (ICAL) to establish a calibration curve for the quantification of the analytes of interest. Obtain secondary review before using the new ICAL.
- 11.1.3. Establish Retention Time Window position for each analyte every after ICAL for proper qualitative identification.
- 11.1.4. Perform Initial Calibration Verification (ICV) every after ICAL to verify accuracy of ICAL.
- 11.1.5. Perform Continuing Calibration Verification (CCV) every 12 hours to verify that the instrument response is reliable, and has not changed significantly from the current ICAL curve.
- 11.1.6. Evaluate Relative Retention Time for each analytes in every sample to be within  $\pm 0.06$  RRT units.
- 11.1.7. Verify Internal Standards (IS) for quantitatively accuracy and that its Retention Time is within  $\pm 30$  seconds from retention time of the midpoint standard in the ICAL and EICP area is within -50 to +100% of ICAL midpoint standard.
- 11.1.8. Evaluate Surrogate recovery to monitor instrument response on every sample.

**11.2. Sample Preparation Batch QC**

- 11.2.1. Analyze Method Blank (MB) to demonstrate that preparation of sample was free from contamination.
- 11.2.2. Analyze Lab Control Sample (LCS) to assess preparative batch accuracy and precision.
- 11.2.3. Analyze Matrix Spike (MS/MSD) to assess matrix interference.
- 11.2.4. Properly treat lab wares used in the sample preparation as specified in EMAX-QC07.
- 11.2.5. Solvents and reagents must undergo quality control check in the stationary laboratory prior to its use. Refer to EMAX-QC01 for details.
  - Verify that the spike amount added is accurate by checking the record.

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- If LCS is within acceptance criteria and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the probable matrix interference in the case narrative.

**11.3. Method QC**

- 11.3.1. All analytes reported must have a valid DL, LOD and LOQ as described in EMAX-QA04.
- 11.3.2. All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.
- 11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency and acceptance criteria.

**12.0 CORRECTIVE ACTIONS**

12.1. Corrective action for each Quality Control Procedures is summarized in Appendix 1.

**12.2. Sample Preparation QC**

- 12.2.1. If the instrument blank is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.1.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new autosampler syringe.
  - 12.2.1.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
  - 12.2.1.3. Rule out preparation contamination by preparing a new instrument blank.
  - 12.2.1.4. If the problem persists, inform the supervisor for further advice.
- 12.2.2. If method blank is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.2.1. Rule out instrument contamination by checking instrument blank.
  - 12.2.2.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01.
  - 12.2.2.3. Rule out glassware contamination used for extraction as described in EMAX-QC07.
  - 12.2.2.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents.
  - 12.2.2.5. If problem persists, inform the supervisor for further advice.
- 12.2.3. If LCS is non-compliant, consider the following suggestions to correct the problem:
  - 12.2.3.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level.
  - 12.2.3.2. If LCS check is within 80-120% of expected value, check the calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.
  - 12.2.3.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS standard, re-extract and re-analyze LCS and the associated samples.

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12.2.3.4. If LCS is within acceptance then and the right amount of spike is added into sample then it is indicative of matrix interference. Discuss the probable matrix interference in Case Narrative.

12.2.4. If MS is non-compliant, consider the following suggestions to correct the problem:

12.2.4.1. Verify from the sample preparation log that the spike amount added is correct.

12.2.4.2. If LCS is within acceptance criteria and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the probability of matrix interference in the case narrative.

**12.3. Analytical Batch QC****12.3.1. Tune Check**

12.3.1.1. If tune check is non-compliant, consider the following suggestion to correct the problem:

- Check the instrument settings and make sure that the instrument parameters are properly set-up.
- Check gas flow.
- Perform auto-tune or visual optimization.
- If the problem persists, inform the supervisor for further actions.

12.3.1.2. If instrument performance is non-compliant, consider the following suggestion to correct the problem:

- Excessive degradation of DDT and/or poor chromatography demonstrated by too much tailing are indications of dirty injection port. Clean or replace the injection port. If problem persist, cut off the first 6-12 inches of the capillary column.

**12.3.2. Initial Calibration (ICAL)**

12.3.2.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

- If RSD% is out of acceptance criteria, review result and identify presence of an outlier.
- If one of the standard return a bias-low or bias-high on all of the analytes, then the point is considered an outlier, prepare a standard at the ICAL point and re-analyze.
- If the highest ICAL point appears to be saturated, drop the highest point.
- If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

*Note: The lowest calibration identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.*

12.3.2.2. If instrumentation problem is suspected, consider the following suggestions to correct the problem:

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- Check the connections and make sure they are air-tight and perform maintenance as needed.
- Check the gas flow.
- Re-tune the MS.
- Prepare a fresh standard and repeat calibration.
- Clean the MS source and repeat the calibration.

12.3.2.3. If the problem persists, inform the supervisor for further action.

12.3.3. Initial Calibration Verification (ICV)

12.3.3.1. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Re-analyze ICV to rule out poor injection.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If problem persist, inform the supervisor for further action.

12.3.4. Daily Calibration Check (DCC)

12.3.4.1. If DCC is non-compliant consider the following suggestions to correct the problem:

- If majority of the analyte response are low and no evidence of leak in the system is apparent, it is indicative of a poor injection or leak in the vial. Re-analyze DCC.
- If problem persist, rule out standard degradation. Prepare a fresh standard and repeat DCC.

12.3.4.2. If Continuing Calibration is non-compliant consider the following suggestions to correct the problem:

- Change the liner.
- Clean the injection port.
- Prepare a new standard.
- Cut or replace column.
- Rule out leaks by checking all connections.
- If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL.

12.3.5. Instrument Blank: If instrument blank is non-compliant, consider the following suggestions to correct the problem:

- Rule out instrument contamination.

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- Rule out reagent contamination by testing each reagent used as described in EMAX-QC01.
  - Rule out vials and glassware contamination as described in EMAX-QC07.
  - If the problem persists, inform the supervisor for further advice.
- 12.4. Discuss in the case narrative, water samples that have residual chlorine above acceptable limits.
- 12.5. Execute a Non-Conformance Report (NCR) when the following circumstances occur:
- 12.5.1. If corrective action needs the function of the department; e.g. if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.
- 12.5.2. If corrective action needs the assistance of the project manager; e.g. if the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.
- 12.6. For other problems encountered, inform the supervisor immediately for further instruction.

**13.0 POLLUTION PREVENTION**

- 13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.
- 13.2. Prepare all standards in fume hoods.

**14.0 WASTE MANAGEMENT**

- 14.1. No samples shall be dumped on the laboratory sink.
- 14.2. Separate and properly identify all unused and expired analytical standards for proper disposal.
- 14.3. Place all waste generated during analytical process in properly labeled satellite waste containers for proper collection.
- 14.4. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

- 15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.1.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples

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originating from various matrices, preparation batches, and can exceed 20 samples.

- 15.1.3. Detection Limit (DL) – The lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not false positive.
- 15.1.4. Limit of Detection (LOD) – An estimate of the minimum amount of substance that an analytical process can reliably detect.
- 15.1.5. Limit of Quantitation (LOQ) – The minimum levels, concentrations or quantities of target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
- 15.1.6. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.
- 15.1.7. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8. Carry-over – are contaminants retained in the instruments/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).
- 15.1.9. Calibration Check Compounds (CCC) – evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.
- 15.1.10. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.11. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.12. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.13. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.14. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.15. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.16. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.17. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.18. Matrix – is a component or form of a sample.
- 15.1.19. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.

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- 15.1.20. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
  - 15.1.21. Response Factor – is the ratio of the peak area of the target compound in the sample or sample extract.
  - 15.1.22. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.
  - 15.1.23. System Performance Check Compounds (SPCC) – are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.

**15.2. Application of QC Procedures**

- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Semi Volatile analysis by GC/MS unless otherwise other directive is specified by the project requirements.

**15.3. Department of Defense (DoD) and Department of Energy Projects**

- 15.3.1. Samples from DoD and DoE sponsored projects follows the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, is applied.

**16.0 REFERENCES**

- 16.1. "Test Methods for Evaluation of Solid Wastes", EPA SW846, Method 8270C as updated
- 16.2. EMAX Quality Systems Manual, as updated

**17.0 APPENDICES****17.1. Tables**

- 17.1.1. Table 1 Intermediate Standard Preparation
  - A) Primary Source B) Secondary Source
- 17.1.2. Table 2A Working Standard Preparation
- 17.1.3. Table 2B Working Standard Preparation for Benzidine
- 17.1.4. Table 2C Working Standard Preparation for Additional Analytes (Appendix IX)
- 17.1.5. Table 3A Working Secondary Source Standard
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- 17.1.8. Table 4 Working Internal Standard Preparation
- 17.1.9. Table 5 Working GC/MS Tuning (DFTPP) Standard
- 17.1.10. Table 6 Instrument Parameters

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- 17.1.11. Table 7 DFTPP Key Ions and Ion Abundance Criteria for 8270C
- 17.1.12. Table 8 Typical Analyte List, Quantitation Ions, IS, Surrogates, Calibration Standards and Detection Limits
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- 17.3. **Appendices**
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- 17.4. **Forms**

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- 17.4.1. 8270FS Sample Preparation Log
- 17.4.2. 8270FA Analytical Run Log
- 17.4.3. 8270FM Instrument Maintenance Log

**Table 1: INTERMEDIATE STANDARD PREPARATION**

**A. Primary Source: Restek, CPI, AccuStandard or equivalent**

Standard Name	Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)	
			Aliquot (µL)	Dil. Solution	Final Vol. (mL)		
Standard List	8270 Mega Mix	1000	Restek	1000	MeCl2	5	200
	8270 Custom Std.	2000	CPI	500	MeCl2	5	200
	Benzidine Mix	2000	Restek	500	MeCl2	5	200
	Benzoic Mix	2000	Restek	500	MeCl2	5	200
	Acid Surrogate Mix	7500	Restek	133.3	MeCl2	5	200
	Base/Neutral Surrogate Mix	5000	Restek	200	MeCl2	5	200
Appendix IX	Appendix IX Mix # 1	2000	Restek	300	MeCl2	5	120
	Appendix IX Mix # 2	1000	Restek	600	MeCl2	5	120
	EPA 8270 Organophosphorous Pesticides Mix	2000	Restek	300	MeCl2	5	120
	Cust. 8270 Std.	2000	AccuStandard	300	MeCl2	5	120
	Acid Surrogate Mix	7500	Restek	80	MeCl2	5	120
	Base/Neutral Surrogate Mix	5000	Restek	120	MeCl2	5	120

**B. Secondary Source: CPI, AccuStandard, Restek or equivalent**

Standard Name	Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)	
			Aliquot (µL)	Dil. Solution	Final Vol. (mL)		
Standard List	Custom Semi Volatile Standard	2000	CPI	NA	NA	NA	NA
	8270 LCS Solution	200	CPI				
	8270 Internal Standard	2000	AccuStandard				
	Benzidine	5000	Ultra				
Appendix IX	Appendix IX Mix # 1*	2000	Restek	300	MeCl2 / MeOH	5	120
	Appendix IX Mix # 2*	1000	Restek	600	MeCl2 / MeOH	5	120
	EPA 8270 Organophosphorous Pesticides Mix*	2000	Restek	300	MeCl2 / MeOH	5	120
	Cust. 8270 Std.	2000	CPI	300	MeCl2 / MeOH	5	120

\*Different Lot

**Table 1 (cont.): INTERMEDIATE STANDARD PREPARATION**

**a. Primary Source: Restek, SPEXertificate, AccuStandard, Ultra Scientific or equivalent**

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Standard List	Benzidine	1000	Restek	NA	NA	NA	NA
Appendix IX Additional	Kepone	2000	SPEXertificate	750	MeCl <sub>2</sub>	5	300
	Famphur	2000	AccuStandard	750	MeCl <sub>2</sub>	5	300
	Hexachloropene	5000	Ultra Scientific	300	MeCl <sub>2</sub>	5	300

**b. Secondary Source: CPI, Supelco or equivalent**

Standard Name		Stock / Internal Soln. Conc. (µg/mL)	Source	Preparation			Final Conc. (µg/mL)
				Aliquot (µL)	Dil. Solution	Final Vol. (mL)	
Appendix IX Additional	Kepone	2000	CPI	750	MeCl <sub>2</sub> / MeOH	5	300
	Famphur	2000	CPI	750	MeCl <sub>2</sub> / MeOH	5	300
	Hexachloropene	5000	Supelco	300	MeCl <sub>2</sub> / MeOH	5	300

**Table 2A: INITIAL CALIBRATION  
WORKING STANDARD PREPARATION**

Standard Name	Intermediate Standard μL of 200 mg/L	Internal Standard μL of 2000 mg/L	Dil. Solvent	Final Volume (μL)	Final Conc.
1	10	10	MeCl <sub>2</sub>	500	4 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
2	25	10	MeCl <sub>2</sub>	500	10 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
3	50	10	MeCl <sub>2</sub>	500	20 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
4	62.5	10	MeCl <sub>2</sub>	500	25 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
5	75	10	MeCl <sub>2</sub>	500	30 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
6	100	10	MeCl <sub>2</sub>	500	40 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
7	125	10	MeCl <sub>2</sub>	500	50 μg/mL of Cal. Std. 40 μg/mL of Internal Std.

**Table 2B: INITIAL CALIBRATION  
WORKING STANDARD PREPARATION FOR BENZIDINE**

Standard Name	Intermediate Standard μL of 1000 mg/L	Internal Standard μL of 2000 mg/L	Dil. Solvent	Final Volume (μL)	Final Conc.
1	2	10	MeCl <sub>2</sub>	500	4 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
2	5	10	MeCl <sub>2</sub>	500	10 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
3	12.5	10	MeCl <sub>2</sub>	500	25 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
4	20	10	MeCl <sub>2</sub>	500	40 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
5	25	10	MeCl <sub>2</sub>	500	50 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
6	40	10	MeCl <sub>2</sub>	500	80 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.
7	50	10	MeCl <sub>2</sub>	500	100 μg/mL of Benzidine Std. 40 μg/mL of Internal Std.

**Table 2C: INITIAL CALIBRATION  
 WORKING STANDARD PREPARATION  
 FOR ADDITIONAL ANALYTES (APPENDIX IX)**

**Appendix IX Standard List**

Standard Name	Intermediate Standard μL of 120 mg/L	Internal Standard μL of 2000 mg/L	Dil. Solvent	Final Volume (μL)	Final Conc.
1	16.7	10	MeCl <sub>2</sub>	500	4 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
2	41.6	10	MeCl <sub>2</sub>	500	10 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
3	83.3	10	MeCl <sub>2</sub>	500	20 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
4	104.2	10	MeCl <sub>2</sub>	500	25 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
5	125	10	MeCl <sub>2</sub>	500	30 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
6	166.7	10	MeCl <sub>2</sub>	500	40 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
7	208.3	10	MeCl <sub>2</sub>	500	50 μg/mL of Cal. Std. 40 μg/mL of Internal Std.

**Appendix IX Additional**

Standard Name	Intermediate Standard μL of 300 mg/L	Internal Standard μL of 2000 mg/L	Dil. Solvent	Final Volume (μL)	Final Conc.
1	33.3	10	MeCl <sub>2</sub>	500	20 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
2	66.6	10	MeCl <sub>2</sub>	500	40 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
3	83.3	10	MeCl <sub>2</sub>	500	50 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
4	133.3	10	MeCl <sub>2</sub>	500	80 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
5	166.6	10	MeCl <sub>2</sub>	500	100 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
6	200	10	MeCl <sub>2</sub>	500	120 μg/mL of Cal. Std. 40 μg/mL of Internal Std.
7	233.3	10	MeCl <sub>2</sub>	500	140 μg/mL of Cal. Std. 40 μg/mL of Internal Std.

**Table 3A: WORKING SECONDARY SOURCE STANDARD**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Custom Semi Volatile Standard	2000	6.25	MeCl <sub>2</sub>	500	25
8270 LCS Solution	200	62.5	MeCl <sub>2</sub>	500	25
8270 Internal Standard	2000	10	MeCl <sub>2</sub>	500	40

**Table 3B: WORKING SECONDARY SOURCE STANDARD  
FOR BENZIDINE**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Benzidine	5000	2.5	MeCl <sub>2</sub>	500	25
8270 Internal Standard	2000	10	MeCl <sub>2</sub>	500	40

**Table 3C: WORKING SECONDARY SOURCE STANDARD  
FOR ADDITIONAL ANALYTES APPENDIX IX**

**Appendix IX Standard List**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Appendix IX Spike	120	104.2	MeCl <sub>2</sub>	500	25
8270 Internal Standard	2000	10	MeCl <sub>2</sub>	500	40

**Appendix IX Additional**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
Appendix IX Add. Spike	300	83.3	MeCl <sub>2</sub>	500	50
8270 Internal Standard	2000	10	MeCl <sub>2</sub>	500	40

**Table 4: WORKING INTERNAL STANDARD PREPARATION**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (mL)	Dil. Solution	Final Vol. (mL)	
Internal Standard Mix	4000	5	MeCl <sub>2</sub>	10	2000

**Table 5: WORKING GC/MS TUNING (DFTPP) STANDARD**

Standard Name	Soln. Conc. (µg/mL)	Preparation			Final Conc. (µg/mL)
		Aliquot (µL)	Dil. Solution	Final Vol. (µL)	
GC/MS Tuning Mix	1000	25	MeCl <sub>2</sub>	500	50

**Table 6: INSTRUMENT PARAMETERS**

	Inst. E4 / Inst. E7	Inst. E9 / Inst. F0
Carrier Gas	Helium at 100 psi at outlet	Helium at 100 psi at outlet
Column head pressure	15 - 35 psi at 40°C	15-35 psi at 45°C
Injection port temperature	280-300°C	200-250°C
Interface	Direct column interference at 280-300°C	Direct column interference at 280-300°C
Valve time	Split 0.2 minute	Split 0.2 minutes
<b>Oven Temperature Program</b>		
Initial Temperature	50°C/min; hold for 0min.	45°C/min; hold for 0.2min.
Rate	30°C/min to 100°C; hold for 0.0 min.; 20°C/min to 200°C; hold for 0.0 min.; 25°C/min to 320°C; hold for 2.53 min.	20°C/min to 320°C; hold for 4 min.
Run Time	14 minutes	18 minutes
<b>Scan Parameters</b>		
Scan start time	After solvent peak	After solvent peak
Mass range	40 to 550 AMU	40 to 550 AMU
Multiplier voltage	1000 - 3000	1000 - 3000
Number of sampling rate	1 - 2	1 - 2
Threshold	0 - 300	0 - 300
Tuning File	DFTPP	DFTPP

**Table 7: DFTTPP KEY IONS ABUNDANCE CRITERIA  
FOR 8270C**

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<b>Mass</b>	<b>Ion Abundance Criteria</b>
51	30 – 60% of mass 198
68	< 2.0% of mass 69
69	Present
70	< 2.0% of mass 69
127	40 – 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 – 30% of mass 198
365	> 1% of mass 198
441	Present but < mass 443
442	> 40% of mass 198
443	17 – 23% of mass 442

**Table 8: TYPICAL TARGET ANALYTE LIST**

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
Acenaphthene	153	152	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Acenaphthylene	152	151	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Aniline	93	66	IS1	Sur1/2	4	10	20	25	40	50			25	40	5.3	10	20	µg/L	83	167	667	µg/Kg
Anthracene	178	152	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Azobenzene*	77	105	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	96	167	333	µg/Kg
Benzo(a)anthracene	228.1	113	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(a)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(b)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	86	167	333	µg/Kg
Benzo(e)pyrene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzo(g,h,i)perylene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
Benzo(k)fluoranthene	252.1	124.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzoic acid	105	77	IS2	Sur3	4	10	20	25	40	50	80	100	25	80	10	20	40	µg/L	333	667	1333	µg/Kg
Benzyl alcohol	108	79	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Biphenyl	154	153	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethoxy) methane	92.9	94.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-Chloroethyl) ether	92.9	62.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis (2-chloroisopropyl) ether	45	76.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Bis(2-ethylhexyl)adipate	129	57	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	87	167	333	µg/Kg
bis(2-Ethylhexyl)phthalate	149	167	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	115	167	333	µg/Kg
4-Bromophenyl-phenylether	247.9	250	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	90	167	333	µg/Kg
Butylbenzylphthalate	149	91	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Carbazole	167	139	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	89	167	333	µg/Kg
4-Chloro-3-methylphenol	107	77	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chloroaniline	127	129	IS2	Sur3	4	10	20	25	40	50			25	40	4.2	5	10	µg/L	83	167	333	µg/Kg
2-Chloronaphthalene	162	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Chlorophenol	127.9	64	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Chlorophenyl-phenylether	204	206	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Chrysene	228.1	226.1	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzo(a,h)anthracene	278.1	139	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dibenzofuran	168	139	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,2-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Dichlorobenzene	145.9	147.9	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3,3'-Dichlorobenzidine	252	254	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	84	167	333	µg/Kg
2,4-Dichlorophenol	161.9	163.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diethylphthalate	149	177	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dimethylnaphthalene	156	141	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	40	50			25	40	2.6	5	10	µg/L	83	167	333	µg/Kg

**Table 8: TYPICAL TARGET ANALYTE LIST**

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
3,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dimethylphthalate	163	164	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-butylphthalate	149	150	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg
4,6-Dinitro-2-methylphenol	198	121	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dinitrobenzene	168	75	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,4-Dinitrophenol	184	106.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
2,4-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dinitrotoluene	165	89	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Di-n-octylphthalate	149	150	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	97	167	333	µg/Kg
Fluoranthene	202	100.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	126	167	333	µg/Kg
Fluorene	166	165	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobenzene	283.8	141.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorobutadiene	224.8	222.8	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachlorocyclopentadiene	236.8	234.8	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachloroethane	116.8	200.8	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Indeno(1,2,3-cd)pyrene	276.1	137.9	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isophorone	82	138	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylnaphthalene	142	141	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Methylphenanthrene	192.1	165	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Methylphenol	107	108	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Naphthalene	128	129	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitroaniline	65	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitroaniline	138	92	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	120	167	333	µg/Kg
Nitrobenzene	77	123	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Nitrophenol	139	65	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
4-Nitrophenol	109	139	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	106	167	667	µg/Kg
N-nitrosodimethylamine	74	42	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitroso-di-n-propylamine	70	42	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-Nitrosodiphenylamine	169	168	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	153	167	333	µg/Kg
Pentachlorophenol	265.8	164.9	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	667	µg/Kg
Perylene	252.1	125	IS6	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	86	167	333	µg/Kg
Phenanthrene	178	152	IS4	Sur5	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Phenol	94	65	IS1	Sur1/2	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Pyrene	202	100.9	IS5	Sur6	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	160	167	333	µg/Kg
Pyridine	79	52	IS1	Sur1/2	4	10	20	25	40	50	80	100	25	80	11	20	40	µg/L	333	667	1333	µg/Kg
2,3,4,6-Tetrachlorophenol	231.8	229.8	IS3	Sur4	4	10	20	25	40	50			25	40	3.6	5	10	µg/L	100	167	333	µg/Kg

**Table 8: TYPICAL TARGET ANALYTE LIST**

Analyte	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)								ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7	8			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
1,2,4-Trichlorobenzene	179.9	181.9	IS2	Sur3	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,4-Trichlorophenol	195.9	96.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,5-Trichlorophenol	195.9	96.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	165	167	333	µg/Kg
2,4,5-Trichlorophenol	195.9	197.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	91	167	333	µg/Kg
2,4,6-Trichlorophenol	195.9	197.9	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,3,5-Trimethylnaphthalene	171.1	155	IS3	Sur4	4	10	20	25	40	50			25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Benzidine	184	92	IS1/4	Sur5	4	10	25	40	50	80	100		25	80	10	20	50	µg/L	863	867	1333	µg/Kg
2-Fluorophenol (Sur1)	112	64	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	61	100	167	µg/Kg
Phenol-d5 (Sur2)	99	71	IS1		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	52	100	167	µg/Kg
Nitrobenzene-d5 (Sur3)	82	128	IS2		4	10	20	25	40	50			25	40	0.59	1	1.67	µg/L	17	33	56	µg/Kg
2-Fluorobiphenyl (Sur4)	172	171	IS3		4	10	20	25	40	50			25	40	0.5	1	1.67	µg/L	24	33	56	µg/Kg
2,4,6-Tribromophenol (Sur5)	329.8	331.8	IS4		4	10	20	25	40	50			25	40	1.5	3	5	µg/L	55	100	167	µg/Kg
Terphenyl-d14 (Sur6)	244.2	122	IS5		4	10	20	25	40	50			25	40	0.54	1	1.67	µg/L	24	33	56	µg/Kg
1,4-Dichlorobenzene-d4 (IS1)	151.9	115																				
Naphtalene-d8 (IS2)	136	68																				
Acenaphthene-d10 (IS3)	164.1	162.1																				
Phenanthrene-d10 (IS4)	188.1	94																				
Chrysene-d12 (IS5)	240.1	120																				
Perylene-d12 (IS6)	264.1	130																				

\*Dissociated to 1,2-Diphenylhydrazine

**Table 9: TYPICAL TARGET ANALYTE LIST FOR APPENDIX IX**

Appendix IX Compound	Quantitation Ions		IS	SUR	ICAL ANALYTE CONCENTRATIONS (µg/L)							ICV/DCC EV(µg/L)	LCS/MS EV(µg/L)	WATER				SOIL			
	Primary	Secondary			1	2	3	4	5	6	7			DL	LOD	LOQ	Unit	DL	LOD	LOQ	Unit
a,a-dimethylphenethylamine	58	91	IS2	Sur3	4	10	20	25	30	40	50	25	40	13	20	40	µg/L	NA	NA	NA	µg/Kg
Acetophenone	77	120	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Acetylaminofluorene	181	223.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.7	5	10	µg/L	83	167	333	µg/Kg
4-Aminobiphenyl	169	115	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	85	167	333	µg/Kg
Aramite	184.9	62.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Atrazine	200	215	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Biphenyl	154	76	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Chlorobenzilate	251	138.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Chloronaphthalene	162	127	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diallate	86	234	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	100	167	333	µg/Kg
Dibenzo(a,j)acridine	279.1	139.3	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2,6-Dichlorophenol	161.9	63	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dimethoate	86.9	124.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	3	5	10	µg/L	89	167	333	µg/Kg
p-Dimethylaminoazobenzene	120	225.1	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
7,12-Dimethylben(a)anthracene	256.1	241.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
3,3-Dimethylbenzidine	212.1	196	IS5	Sur6	4	10	20	25	30	40	50	25	40	5	10	20	µg/L	130	167	667	µg/Kg
3,4-Dimethylphenol	107	122	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,3-Dinitrobenzene	167.9	75.9	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Dinoseb	211	162.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Dioxane	88	57.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Diphenyl ether	170	141	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Disulfoton	88	96.9	IS4	Sur5	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl methacrylate	69	41	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl methanesulfonate	78.9	108.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Ethyl parathion	96.9	108.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Hexachloropropene	212.8	140.8	IS2	Sur3	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isodrin	192.9	262.8	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Isosafrole	162	104	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methapyrilene	58	96.9	IS5	Sur6	4	10	20	25	30	40	50	25	40	10	20	40	µg/L	NA	NA	NA	µg/Kg
3-Methylcholanthrene	268.1	252.1	IS6	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methyl methanesulfonate	79.9	78.9	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
Methyl parathion	108.9	263	IS5	Sur6	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1,4-Naphthoquinone	158	130	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
1-Naphthylamine	143	115	IS3	Sur4	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
2-Naphthylamine	143	115	IS3	Sur4	4	10	20	25	30	40	50	25	40	10	10	20	µg/L	330	333	667	µg/Kg
N-nitrosodiethylamine	102	56	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg
N-nitrosomethylethylamine	88	42	IS1	Sur1/2	4	10	20	25	30	40	50	25	40	2.5	5	10	µg/L	83	167	333	µg/Kg



**Table 10: CALIBRATION CHECK COMPOUNDS (CCCs)**

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<b>Base/Neutral Fraction</b>	<b>Acid Fraction</b>
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadine	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

**Table 11: SYSTEM PERFORMANCE CALIBRATION CHECK COMPOUNDS (SPCCs)**

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<b>Base/Neutral Compounds</b>	<b>Acid Compounds</b>
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

**Figure 1:** **PEAK EVALUATION TECHNIQUE**

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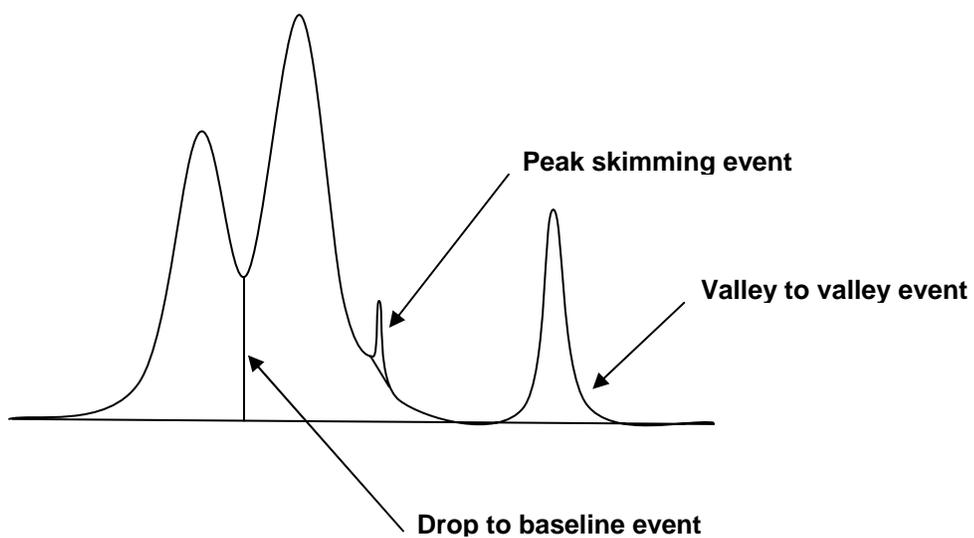
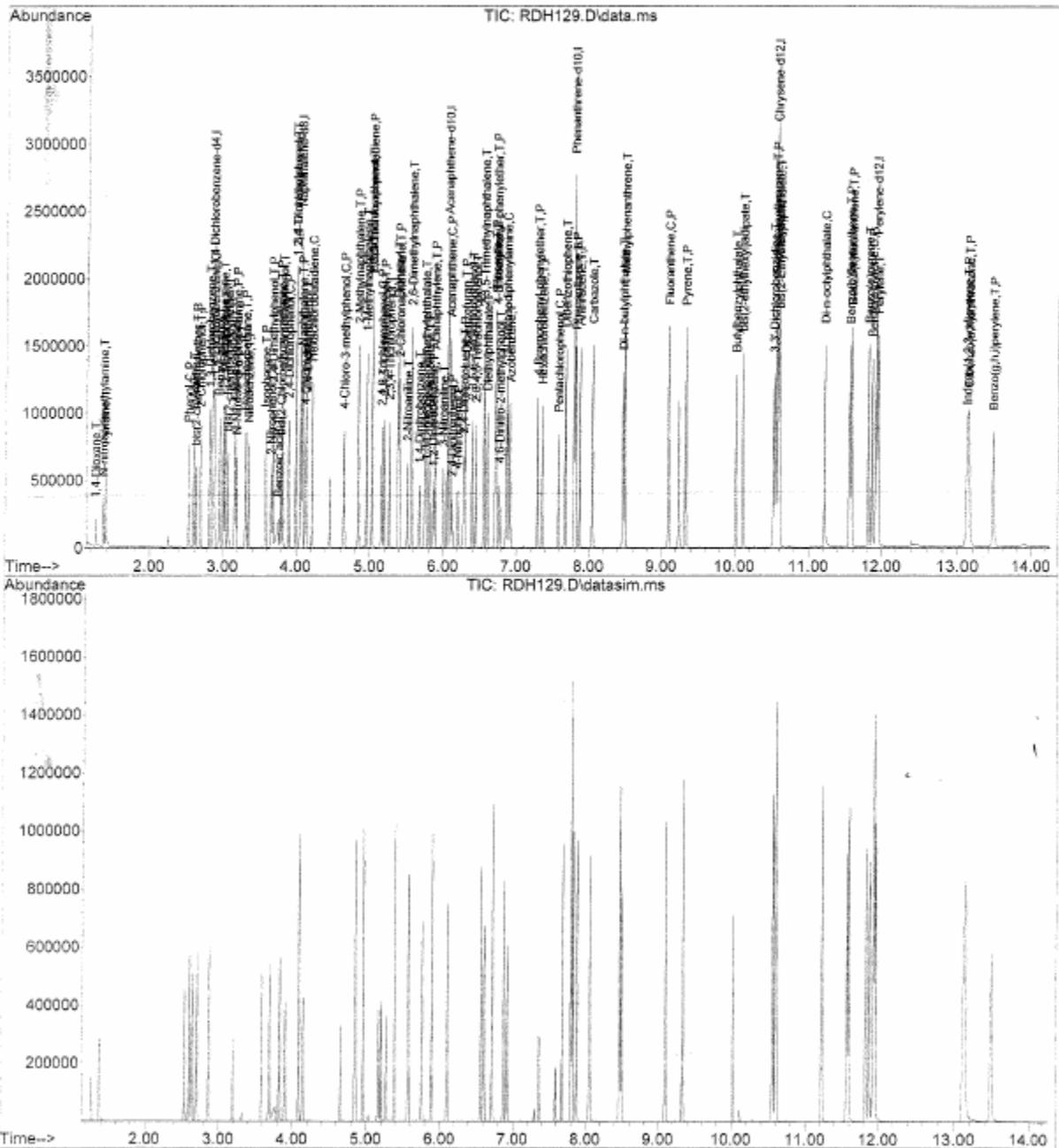


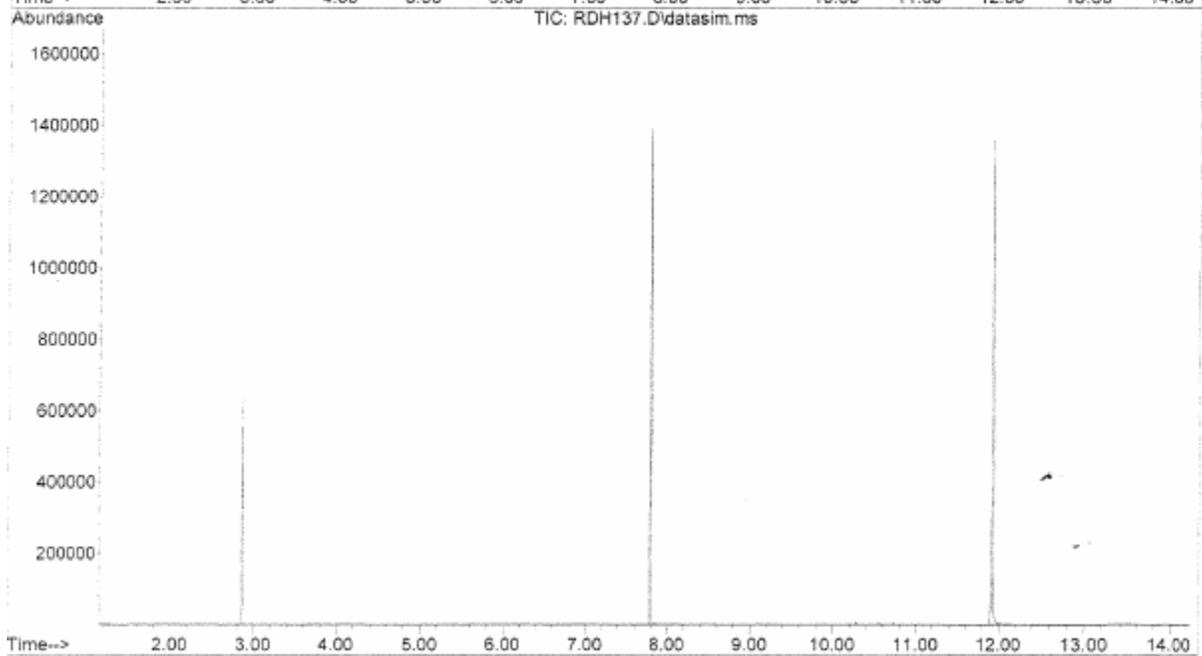
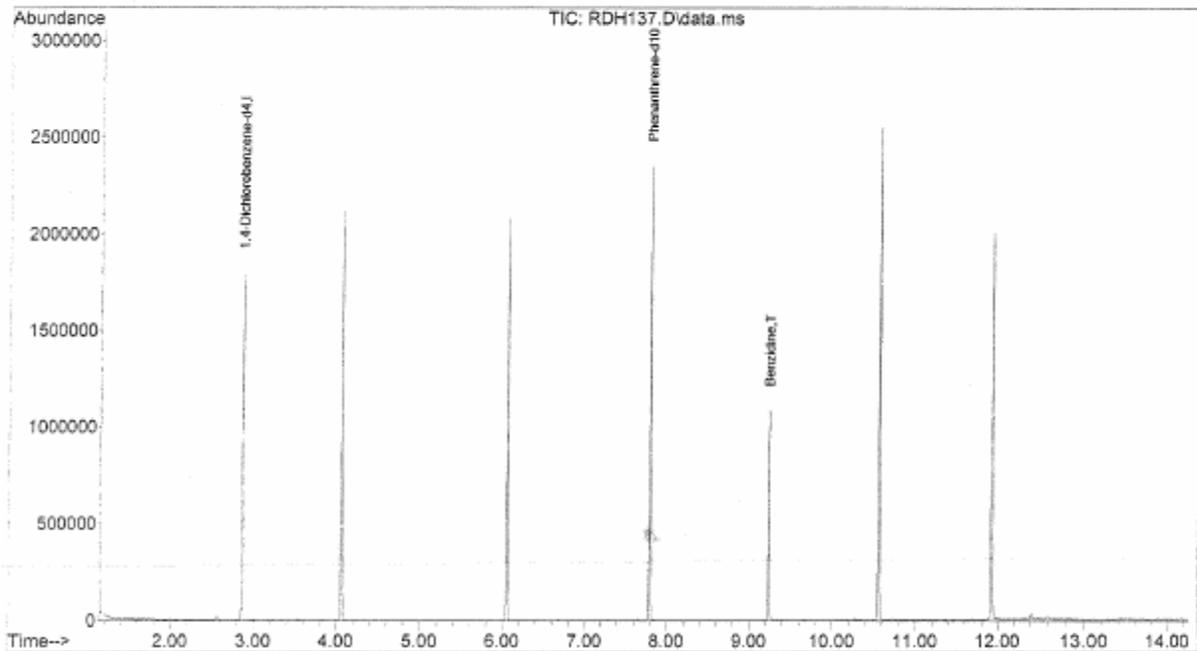
Figure 2: TYPICAL CHROMATOGRAM

Quantitation Report (QT Reviewed)  
Data File : D:\DATA\14D10\RDH129.D Vial: 16  
Acq On : 10 Apr 2014 20:01 Operator: KV  
Sample : ISVE7D101 Inst : E7  
Misc : ICV Multiplr: 1.00  
Integrator: RTE  
Quant Time: Apr 11 11:46:25 2014  
Quant Results File: SVE7D10.RES  
Quant Method : C:\msdchem\1\METHODS\SVE7D10.M  
Quant Title : SEMIVOLATILES  
QLast Update : Thu Apr 10 18:33:28 2014  
Response via : Initial Calibration  
DataAcq Meth: SVE7D10S.M



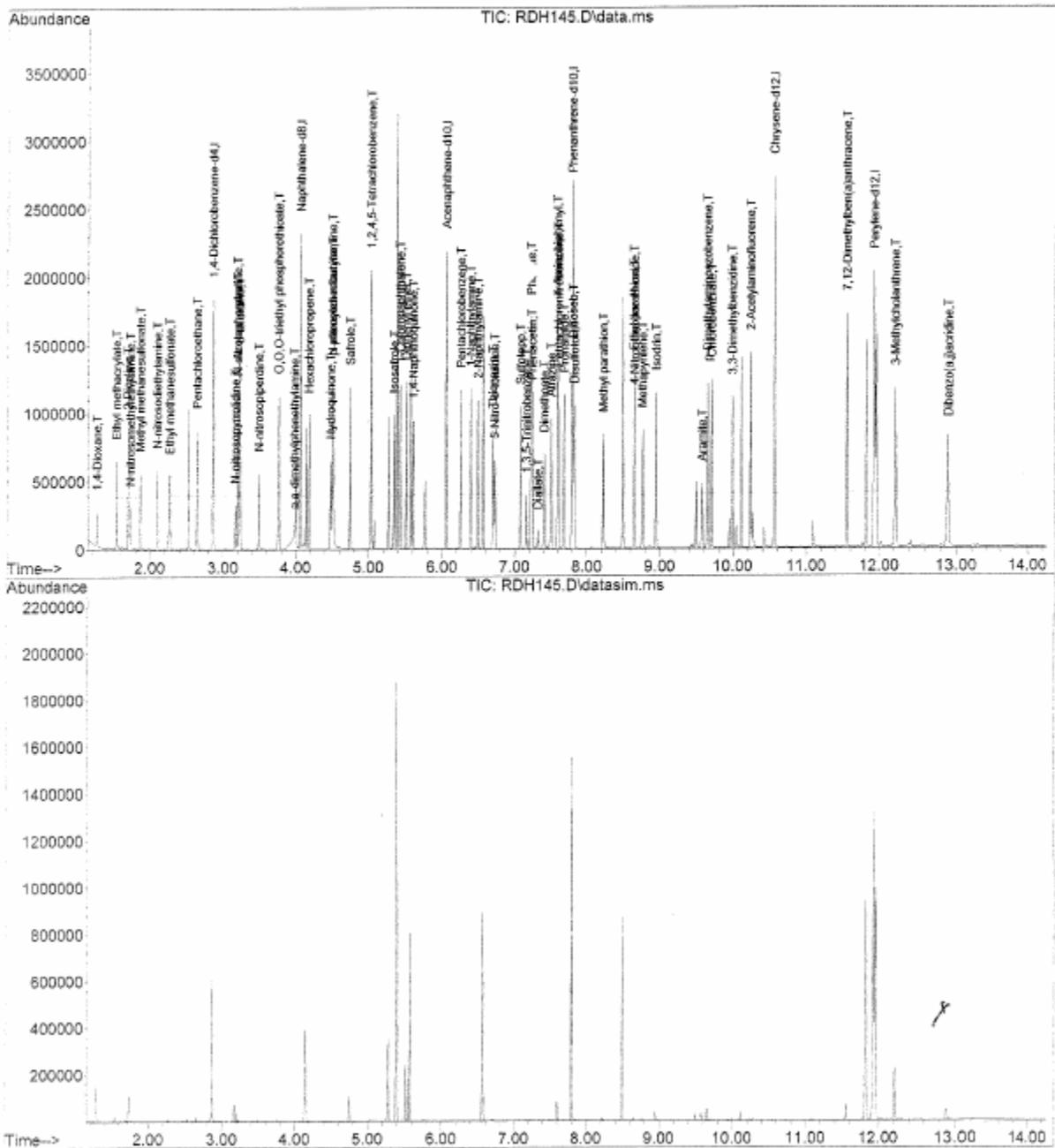
**Figure 2A: TYPICAL CHROMATOGRAM FOR BENZIDINE**

Quantitation Report (QT Reviewed)  
Data File : D:\DATA\14D10\RDH137.D Vial: 24  
Acq On : 10 Apr 2014 22:36 Operator: KV  
Sample : ISVE7D10A1 Inst : E7  
Misc : ICV Multiplr: 1.00  
Integrator: RTE  
Quant Time: Apr 11 11:57:29 2014  
Quant Results File: SVE7D10A.RES  
Quant Method : C:\msdchem\1\METHODS\SVE7D10A.M  
Quant Title : Semivolatiles - Benzidine  
QLast Update : Fri Apr 11 11:56:02 2014  
Response via : Initial Calibration  
DataAcq Meth:SVE7D10S.M



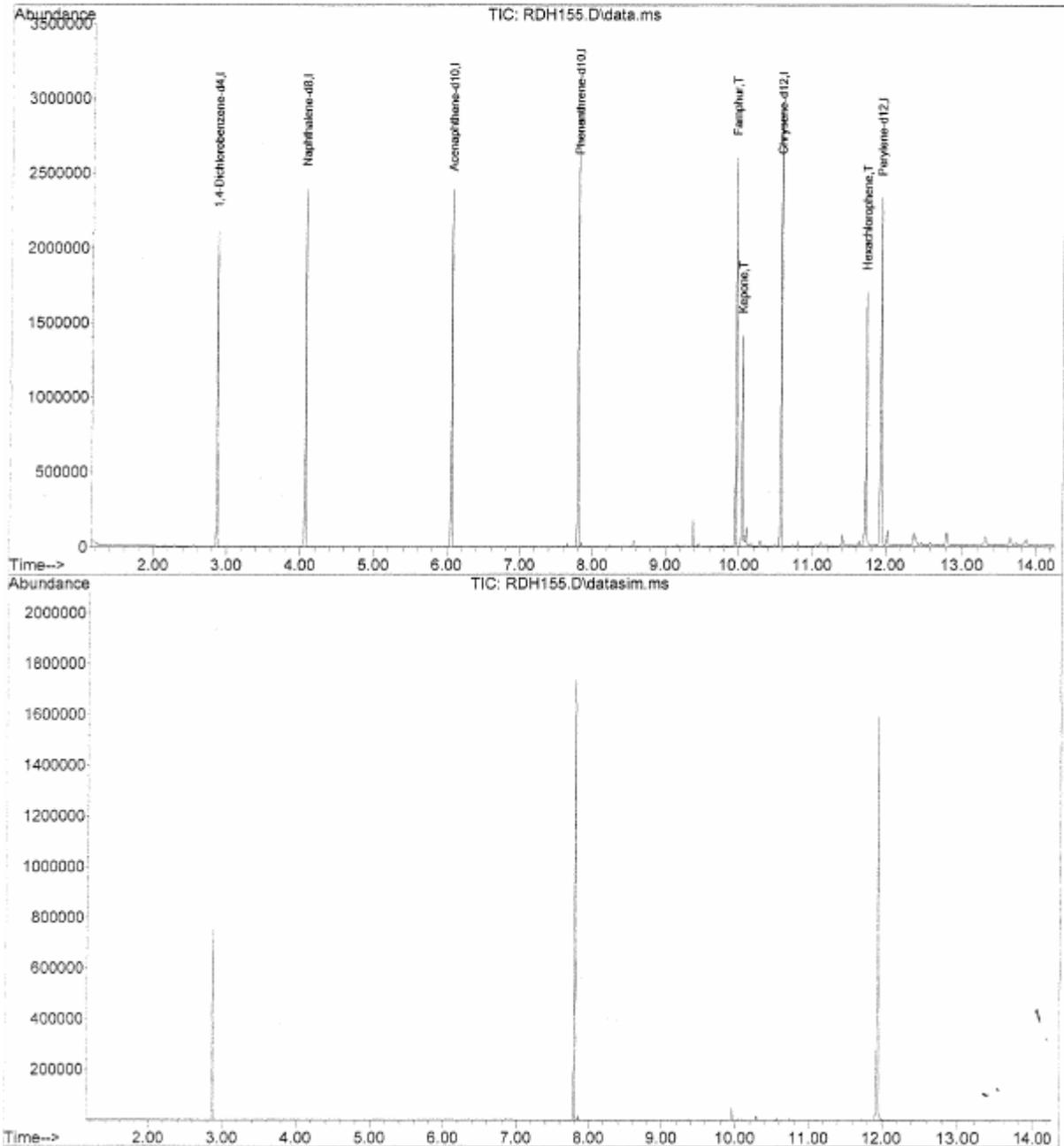
**Figure 2B: TYPICAL CHROMATOGRAM FOR APPENDIX IX**

Quantitation Report (QT Reviewed)  
 Data File : D:\DATA\14D10\RDH145.D Vial: 32  
 Acq On : 11 Apr 2014 1:11 Operator: KV  
 Sample : ISVE7D10B1 Inst : E7  
 Misc : 25PPM Multiplr: 1.00  
 Integrator: RTE  
 Quant Time: Apr 11 12:56:11 2014  
 Quant Results File: SVE7D10B.RES  
 Quant Method : C:\msdchem\1\METHODS\SVE7D10B.M  
 Quant Title : SEMIVOLATILES APP9  
 QLast Update : Fri Apr 11 12:49:28 2014  
 Response via : Initial Calibration  
 DataAcq Meth: SVE7D10S.M



**Figure 2B (cont.): TYPICAL CHROMATOGRAM FOR APPENDIX IX**

Quantitation Report (QT Reviewed)  
Data File : D:\DATA\14D11\RDH155.D Vial: 10  
Acq On : 11 Apr 2014 17:10 Operator: DJ  
Sample : ISVE7D11C1 Inst : E7  
Misc : 50PPM Multiplr: 1.00  
Integrator: RTE  
Quant Time: Apr 11 17:33:29 2014  
Quant Results File: SVE7D11C.RES  
Quant Method : C:\msdchem\1\METHODS\SVE7D11C.M  
Quant Title : SEMIVOLATILES APP9  
QLast Update : Fri Apr 11 17:30:55 2014  
Response via : Initial Calibration  
DataAcq Meth:SVE7D10S.M



**Figure 3: TYPICAL ICAL SUMMARY**

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Instrument ID :E4  
Beginning Date/Time :02/18/14 11:45  
Spike Units :PPM  
IC File :RBJ065

Column Spec :Rxi-5SilMS ID :0.18MM  
Ending Date/Time :02/18/14 14:01  
HPCChem Method :SVE4B18

IDX	Parameters	4	10	20	25	40	50	80	100	Av_RRF	%_RSD	Av_Rt_M
		14:01 RBJ068	13:41 RBJ067	13:22 RBJ066	13:03 RBJ065	12:43 RBJ064	12:24 RBJ063	12:05 RBJ062	11:45 RBJ061			
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	1	0	3.0245
2	1,4-Dioxane	0.509	0.390	0.448	0.428	0.413	0.414	-----	-----	0.434	9.62	1.3713
3	N-nitrosodimethylamine	0.599	0.596	0.620	0.603	0.589	0.581	-----	-----	0.598	2.24	1.4806
4	Pyridine	1.163	1.093	1.184	1.121	1.110	1.102	1.158	1.125	1.132	2.90	1.5077
5	2-Fluorophenol	1.029	0.973	1.053	1.029	1.008	1.004	-----	-----	1.016	2.68	2.0861
6	Phenol-d5	1.212	1.238	1.333	1.311	1.294	1.275	-----	-----	1.277	3.55	2.6749
7	Phenol	1.312	1.303	1.394	1.360	1.341	1.342	-----	-----	1.342	2.46	2.6871
8	Aniline	1.681	1.642	1.768	1.712	1.718	1.699	-----	-----	1.704	2.47	2.7514
9	bis(2-chloroethyl)ether	0.986	0.951	1.015	0.998	0.951	0.972	-----	-----	0.979	2.64	2.7749
10	2-Chlorophenol	1.166	1.154	1.226	1.168	1.179	1.173	-----	-----	1.178	2.14	2.8513
11	1,3-Dichlorobenzene	1.413	1.394	1.460	1.416	1.364	1.379	-----	-----	1.404	2.40	2.9813
12	1,2-Dichlorobenzene-d4	0.890	0.888	0.916	0.897	0.883	0.875	-----	-----	0.892	1.58	3.1621
13	1,4-Dichlorobenzene	1.439	1.423	1.507	1.441	1.423	1.413	-----	-----	1.441	2.37	3.0389
14	Benzyl alcohol	0.703	0.707	0.774	0.763	0.753	0.749	-----	-----	0.742	3.99	3.1157
15	1,2-Dichlorobenzene	1.354	1.308	1.406	1.379	1.352	1.356	-----	-----	1.359	2.38	3.1749
16	2-Methylphenol	0.876	0.905	0.976	0.953	0.948	0.941	-----	-----	0.933	3.90	3.1957
17	bis(2-chloroisopropyl)ether	1.904	1.877	1.989	1.909	1.875	1.822	-----	-----	1.896	2.89	3.2341
18	4-Methylphenol	1.172	1.167	1.223	1.226	1.209	1.183	-----	-----	1.197	2.17	3.3364
19	N-Nitroso-di-n-propylamine	0.768	0.766	0.833	0.817	0.798	0.769	-----	-----	0.792	3.60	3.3563
20	Hexachloroethane	0.529	0.548	0.591	0.565	0.564	0.555	-----	-----	0.559	3.69	3.4872
21	Naphthalene-d8	1	1	1	1	1	1	1	1	1	0	4.2527
22	Nitrobenzene-d5	0.421	0.423	0.454	0.397	0.388	0.387	-----	-----	0.412	6.35	3.5128
23	Nitrobenzene	0.374	0.378	0.411	0.396	0.396	0.396	-----	-----	0.392	3.46	3.5303
24	Isophorone	0.608	0.615	0.656	0.707	0.707	0.717	-----	-----	0.668	7.34	3.7598
25	2-Nitrophenol	0.168	0.169	0.186	0.176	0.179	0.180	-----	-----	0.176	3.80	3.8445
26	2,4-Dimethylphenol	0.368	0.368	0.395	0.376	0.377	0.379	-----	-----	0.377	2.66	3.8637
27	Benzoic acid	0.121	0.195	0.238	0.234	0.240	0.274	0.253	0.253	0.226	21.27	3.9613
28	bis(2-Chloroethoxy)methane	0.350	0.359	0.378	0.357	0.367	0.368	-----	-----	0.363	2.77	3.9643
29	3,5-Dimethylphenol	0.360	0.423	0.448	0.427	0.437	0.437	-----	-----	0.422	7.45	4.0026
30	2,4-Dichlorophenol	0.308	0.309	0.338	0.329	0.324	0.327	-----	-----	0.322	3.68	4.0860
31	3,4-Dimethylphenol	0.438	0.444	0.484	0.465	0.471	0.480	-----	-----	0.464	4.03	4.1817
32	1,2,4-Trichlorobenzene	0.401	0.387	0.416	0.403	0.405	0.408	-----	-----	0.403	2.32	4.1873
33	Naphthalene	0.948	0.958	1.031	0.986	0.985	1.002	-----	-----	0.985	3.04	4.2742
34	4-Chloroaniline	0.388	0.396	0.439	0.413	0.415	0.419	-----	-----	0.412	4.36	4.3169
35	2,6-Dichlorophenol	0.306	0.299	0.328	0.316	0.318	0.322	-----	-----	0.315	3.37	4.3305
36	Hexachlorobutadiene	0.283	0.286	0.297	0.283	0.290	0.291	-----	-----	0.288	1.93	4.4118
37	Hydroquinone	-----	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
38	4-Chloro-3-methylphenol	0.261	0.283	0.307	0.301	0.299	0.299	-----	-----	0.292	5.80	4.8467
39	2-Methylnaphthalene	0.661	0.659	0.707	0.678	0.684	0.695	-----	-----	0.680	2.81	5.0423
40	1-Methylnaphthalene	0.614	0.607	0.648	0.632	0.630	0.636	-----	-----	0.628	2.38	5.1573
41	Acenaphthene-d10	1	1	1	1	1	1	1	1	1	0	6.2772
42	Hexachlorocyclopentadiene	0.518	0.512	0.573	0.529	0.553	0.569	-----	-----	0.542	4.83	5.2364
43	2,3,5-Trichlorophenol	0.312	0.332	0.371	0.392	0.370	0.421	-----	-----	0.366	10.81	5.2356
44	2,4,6-Trichlorophenol	0.412	0.424	0.463	0.434	0.446	0.449	-----	-----	0.438	4.19	5.3654
45	2,4,5-Trichlorophenol	0.418	0.430	0.471	0.452	0.469	0.468	-----	-----	0.451	4.99	5.4033
46	2-Fluorobiphenyl	1.449	1.468	1.566	1.696	1.526	1.795	-----	-----	1.583	8.58	5.4693
47	2,3,4-Trichlorophenol	0.422	0.420	0.447	0.429	0.460	0.447	-----	-----	0.437	3.69	5.4789
48	Biphenyl	1.390	1.403	1.543	1.433	1.464	1.497	-----	-----	1.455	4.02	5.5870
49	2-Chloronaphthalene	1.131	1.156	1.243	1.170	1.173	1.202	-----	-----	1.179	3.29	5.6102
50	2-Nitroaniline	0.310	0.332	0.366	0.341	0.345	0.352	-----	-----	0.341	5.56	5.7229
51	2,6-Dimethylnaphthalene	0.993	1.054	1.109	1.045	1.063	1.095	-----	-----	1.060	3.88	5.7741
52	1,4-Dinitrobenzene	0.132	0.151	0.173	0.165	0.166	0.167	-----	-----	0.159	9.39	5.8772
53	Dimethylphthalate	1.511	1.509	1.646	1.511	1.483	1.511	-----	-----	1.528	3.82	5.9517
54	1,3-Dinitrobenzene	0.168	0.180	0.197	0.186	0.188	0.194	-----	-----	0.185	5.64	5.9726
55	2,6-Dinitrotoluene	0.249	0.265	0.284	0.271	0.274	0.272	-----	-----	0.269	4.34	6.0144

Figure 3 (cont.): TYPICAL ICAL SUMMARY

56	1,2-Dinitrobenzene	0.102	0.112	0.120	0.112	0.112	0.116	-----	-----	0.113	5.35	6.0771
57	Acenaphthylene	1.692	1.729	1.881	1.770	1.788	1.812	-----	-----	1.779	3.70	6.1053
58	3-Nitroaniline	0.245	0.265	0.281	0.267	0.268	0.274	-----	-----	0.267	4.63	6.2152
59	Acenaphthene	1.156	1.143	1.234	1.155	1.184	1.195	-----	-----	1.178	2.85	6.3167
60	2,4-Dinitrophenol	0.130	0.156	0.186	0.175	0.189	0.192	-----	-----	0.172	14.10	6.3366
61	4-Nitrophenol	0.203	0.228	0.253	0.230	0.243	0.248	-----	-----	0.234	7.77	6.4165
62	2,4-Dinitrotoluene	0.327	0.363	0.393	0.364	0.363	0.370	-----	-----	0.363	5.78	6.5004
63	Dibenzofuran	1.588	1.632	1.740	1.637	1.672	1.678	-----	-----	1.658	3.11	6.5228
64	2,3,5,6-Tetrachlorophenol	0.287	0.307	0.329	0.306	0.323	0.326	-----	-----	0.313	5.12	6.6194
65	2,3,4,6-Tetrachlorophenol	0.345	0.374	0.405	0.370	0.422	0.441	-----	-----	0.393	9.19	6.6722
66	2,3,5-Trimethylnaphthalene	0.966	0.968	1.046	0.988	1.007	1.034	-----	-----	1.002	3.35	6.7808
67	Diethylphthalate	1.323	1.323	1.422	1.337	1.356	1.380	-----	-----	1.357	2.84	6.8074
68	Fluorene	1.302	1.310	1.422	1.355	1.396	1.439	-----	-----	1.370	4.20	6.9356
69	4-Chlorophenyl-phenylether	0.714	0.720	0.778	0.732	0.746	0.772	-----	-----	0.744	3.61	6.9379
70	4-Nitroaniline	0.244	0.262	0.290	0.262	0.273	0.276	-----	-----	0.268	5.87	6.9535
71	Phenanthrene-d10	1	1	1	1	1	1	1	1	1	0	8.0159
72	4,6-Dinitro-2-methylphenol	0.097	0.123	0.140	0.131	0.135	0.132	-----	-----	0.126	12.28	6.9926
73	N-Nitrosodiphenylamine	0.608	0.578	0.546	0.523	0.524	0.568	-----	-----	0.558	5.95	7.0767
74	Azobenzene	0.598	0.577	0.628	0.603	0.595	0.593	-----	-----	0.599	2.79	7.1258
75	2,4,6-Tribromophenol	0.092	0.093	0.105	0.097	0.098	0.099	-----	-----	0.097	4.74	7.2191
76	4-Bromophenyl-phenylether	0.207	0.203	0.215	0.204	0.206	0.203	-----	-----	0.207	2.23	7.5094
77	Hexachlorobenzene	0.228	0.213	0.228	0.222	0.218	0.214	-----	-----	0.221	3.11	7.5889
78	Pentachlorophenol	0.124	0.140	0.151	0.143	0.145	0.146	-----	-----	0.142	6.58	7.8061
79	Dibenzothiofene	0.884	0.882	0.945	0.987	0.887	1.005	-----	-----	0.932	5.95	7.8997
80	Phenanthrene	0.976	0.966	1.021	0.981	0.970	0.972	-----	-----	0.981	2.07	8.0419
81	Dinoseb	-----	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
82	Anthracene	0.981	0.979	1.040	0.993	0.995	0.993	-----	-----	0.997	2.21	8.0975
83	Carbazole	0.836	0.846	0.895	0.833	0.839	0.841	-----	-----	0.848	2.75	8.2730
84	Di-n-butylphthalate	1.012	1.030	1.122	1.050	1.059	1.068	-----	-----	1.057	3.57	8.6639
85	1-Methylphenanthrene	0.714	0.715	0.777	0.727	0.726	0.731	-----	-----	0.732	3.17	8.7171
86	Fluoranthene	1.111	1.163	1.244	1.173	1.182	1.202	-----	-----	1.179	3.72	9.3297
87	Chrysene-d12	1	1	1	1	1	1	1	1	1	0	10.8160
88	Benzdine	-----	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
89	Pyrene	1.059	1.039	1.100	1.051	1.041	1.028	-----	-----	1.053	2.40	9.5665
90	Terphenyl-d14	0.719	0.701	0.750	0.703	0.705	0.692	-----	-----	0.712	2.91	9.7277
91	Butylbenzylphthalate	0.361	0.377	0.422	0.392	0.392	0.391	-----	-----	0.389	5.21	10.2291
92	bis(2-ethylhexyl)adipate	0.282	0.290	0.317	0.299	0.301	0.292	-----	-----	0.297	4.08	10.3082
93	3,3'-Dichlorobenzidine	0.323	0.348	0.362	0.346	0.347	0.347	-----	-----	0.346	3.58	10.7722
94	Benzo(a)anthracene	1.035	1.013	1.072	1.012	1.024	1.021	-----	-----	1.029	2.18	10.8022
95	Chrysene	0.931	0.928	0.965	0.918	0.918	0.908	-----	-----	0.928	2.14	10.8419
96	bis(2-Ethylhexyl)phthalate	0.602	0.616	0.669	0.639	0.632	0.630	-----	-----	0.631	3.57	10.8127
97	Perylene-d12	1	1	1	1	1	1	1	1	1	0	12.2554
98	Di-n-octylphthalate	0.969	1.019	1.179	1.084	1.117	1.135	-----	-----	1.084	7.19	11.4319
99	Benzo(b)fluoranthene	0.990	1.002	1.130	1.005	1.068	1.105	-----	-----	1.050	5.64	11.8473
100	Benzo(k)fluoranthene	0.966	0.989	1.067	1.056	1.010	1.014	-----	-----	1.017	3.81	11.8754
101	Benzo(e)pyrene	0.933	0.917	1.033	0.953	0.965	0.983	-----	-----	0.964	4.26	12.1343
102	Benzo(a)pyrene	0.923	0.946	1.040	0.973	0.990	1.005	-----	-----	0.980	4.25	12.1910
103	Perylene	0.995	0.986	1.050	0.980	0.984	1.001	-----	-----	0.999	2.60	12.2849
104	Indeno(1,2,3-cd)pyrene	1.069	1.070	1.202	1.112	1.146	1.172	-----	-----	1.129	4.83	13.5858
105	Dibenzo(a,h)anthracene	0.858	0.897	0.995	0.925	0.956	0.981	-----	-----	0.935	5.58	13.5980
106	Benzo(g,h,i)perylene	0.898	0.908	0.989	0.941	0.930	0.950	-----	-----	0.936	3.48	13.9846

Ave\_%RSD : 4.5 Max\_%RSD : 21.3

Use Least Square Linear Regression with weighting factor of inverse concentration  
Resp\_Ratio = x0 + x1 \* Amt\_Ratio

IDX	Parameter	x0	x1	CCF
27	Benzoic acid	-0.01462	0.26295	0.9988

**Figure 3A: TYPICAL ICAL SUMMARY OF BENZIDINE**

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Instrument ID :E4  
 Beginning DateTime :02/18/14 09:10  
 Spike Units :PPM  
 IC File :RBJ057

Column Spec :Rxi-5Si1MS ID :0.18MM  
 Ending DateTime :02/18/14 11:07  
 HPCHEM Method :SVE4B18A

IDX	Parameters	4	10	25	40	50	80	100	Av_RRF	%_RSD	Av_Rt_M
		11:07 RBJ059	10:47 RBJ058	10:28 RBJ057	10:09 RBJ056	09:49 RBJ055	09:30 RBJ054	09:10 RBJ053			
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	Benzaldehyde	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
3	Caprolactam	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
4	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0143
5	Benzidine	0.585	0.692	0.774	0.775	0.778	0.785	0.820	0.744	10.77	9.4674

Ave\_%RSD : 10.8

Max\_%RSD : 10.8

**Figure 3B: TYPICAL ICAL SUMMARY OF APPENDIX IX**

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Instrument ID :E4  
Beginning Date/Time :02/18/14 16:27  
Spike Units :PPM  
IC File :RBJ078

Column Spec :Rxi-5SilMS ID :0.18MM  
Ending Date/Time :02/18/14 18:43  
HPChem Method :SVE4B18B

IDX	Parameters	4	10	20	25	30	40	50	Av_RRF	%_RSD	Av_Rt_M
		18:43 RBJ081	18:24 RBJ080	18:05 RBJ079	17:26 RBJ078	17:07 RBJ077	16:47 RBJ076	16:27 RBJ075			
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	1,4-Dioxane	0.465	0.420	0.478	0.471	0.479	0.469	0.476	0.465	4.39	1.3713
3	Ethyl methacrylate	0.887	0.901	0.938	0.908	0.915	0.907	0.926	0.912	1.82	1.6489
4	2-Picoline	1.242	1.210	1.266	1.222	1.242	1.242	1.239	1.238	1.43	1.8165
5	N-nitrosomethyl ethylamine	0.480	0.475	0.469	0.460	0.473	0.459	0.458	0.468	1.92	1.8507
6	Methyl methanesulfonate	0.794	0.912	0.941	0.908	0.926	0.894	0.899	0.896	5.33	1.9942
7	2-Fluorophenol	1.113	1.068	1.111	1.072	1.088	1.092	1.094	1.091	1.57	2.0894
8	N-nitrosodiethylamine	0.433	0.459	0.484	0.473	0.484	0.479	0.486	0.471	4.09	2.2339
9	Ethyl methanesulfonate	0.937	0.889	0.937	0.904	0.908	0.921	0.913	0.916	1.92	2.4017
10	Phenol-d5	1.416	1.333	1.398	1.399	1.414	1.404	1.413	1.397	2.08	2.6748
11	Benzaldehyde	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
12	Pentachloroethane	0.595	0.635	0.648	0.616	0.633	0.623	0.633	0.626	2.73	2.7992
13	1,2-Dichlorobenzene-d4	0.998	0.990	1.035	0.983	1.005	1.019	1.030	1.009	1.97	3.1638
14	N-nitrosopyrrolidine	0.392	0.420	0.480	0.456	0.469	0.469	0.454	0.449	6.99	3.3351
15	Naphthalene-d8	1	1	1	1	1	1	1	1	0	4.2541
16	Acetophenone	0.446	0.435	0.448	0.441	0.448	0.444	0.451	0.445	1.22	3.3645
17	N-nitrosomorpholine	0.237	0.236	0.248	0.246	0.248	0.247	0.247	0.244	2.18	3.3652
18	o-toluidine	0.537	0.608	0.547	0.616	0.552	0.633	0.637	0.590	7.34	3.3994
19	Nitrobenzene-d5	0.462	0.453	0.468	0.467	0.480	0.477	0.472	0.468	1.98	3.5123
20	N-nitrosopiperidine	0.158	0.147	0.156	0.152	0.157	0.157	0.155	0.155	2.38	3.6706
21	O,O,O-triethyl phosphorothioate	0.153	0.151	0.157	0.156	0.157	0.161	0.164	0.157	2.77	3.9336
22	a,a-dimethylphenethylamine	1.090	1.043	1.050	1.030	1.006	0.982	0.914	1.016	5.58	4.1700
23	3,4-Dimethylphenol	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
24	2,6-Dichlorophenol	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
25	Hexachloropropene	0.355	0.356	0.377	0.374	0.381	0.380	0.383	0.372	3.17	4.3755
26	Hydroquinone	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
27	N-nitrosodi-n-butylamine	0.237	0.228	0.303	0.292	0.285	0.289	0.285	0.274	10.65	4.6892
28	p-phenylenediamine	0.279	0.338	0.300	0.290	0.290	0.267	0.256	0.289	9.17	4.6988
29	epsilon-Caprolactam	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
30	Safrole	0.298	0.292	0.304	0.301	0.302	0.306	0.356	0.309	6.95	4.9365
31	1-Methylnaphthalene	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
32	1,2,4,5-Tetrachlorobenzene	0.476	0.459	0.485	0.482	0.489	0.492	0.491	0.482	2.41	5.2417
33	Acenaphthene-d10	1	1	1	1	1	1	1	1	0	6.2771
34	2-Fluorobiphenyl	1.709	1.687	1.768	1.689	1.994	1.998	2.024	1.838	8.63	5.4710
35	Isosafrole	0.530	0.491	0.517	0.498	0.509	0.518	0.519	0.512	2.61	5.5453
36	Biphenyl	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
37	1-Chloronaphthalene	1.168	1.140	1.184	1.159	1.184	1.192	1.203	1.176	1.81	5.6397
38	Diphenyl ether	0.832	0.954	0.866	0.847	0.843	0.852	0.865	0.866	4.74	5.7124
39	1,4-Naphthoquinone	0.402	0.429	0.403	0.373	0.347	0.322	0.316	0.370	11.79	5.8083
40	1,3-Dinitrobenzene	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
41	Pentachlorobenzene	0.647	0.630	0.635	0.638	0.652	0.654	0.657	0.645	1.61	6.4818
42	1-Naphthylamine	1.047	1.127	1.104	1.066	1.099	1.104	1.110	1.094	2.52	6.6099
43	2,3,4,6-Tetrachlorophenol	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
44	2-Naphthylamine	1.248	1.277	1.200	1.153	1.174	1.187	1.198	1.205	3.56	6.7064
45	Thionazin	0.177	0.185	0.183	0.181	0.184	0.184	0.183	0.183	1.38	6.8995
46	5-Nitro-o-toluidine	0.279	0.339	0.320	0.317	0.326	0.320	0.327	0.318	5.89	6.9438
47	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0190
48	2,4,6-Tribromophenol	0.092	0.095	0.100	0.099	0.104	0.100	0.101	0.099	3.94	7.2173
49	Sulfotepp	0.077	0.076	0.084	0.084	0.084	0.085	0.084	0.082	4.54	7.2756
50	1,3,5-Trinitrobenzene	0.049	0.055	0.060	0.061	0.066	0.061	0.063	0.059	9.19	7.3744

**Figure 3B (cont.): TYPICAL ICAL SUMMARY OF APPENDIX IX**

51	Phorate	0.325	0.315	0.351	0.345	0.348	0.343	0.348	0.339	4.02	7.4303
52	Phenacetin	0.298	0.303	0.299	0.297	0.318	0.296	0.306	0.302	2.52	7.4439
53	Diallate	0.037	0.027	0.029	0.027	0.027	0.026	0.026	0.028	13.80	7.5241
54	Dimethoate	0.182	0.181	0.174	0.172	0.175	0.164	0.165	0.173	4.10	7.6166
55	Atrazine	0.208	0.194	0.187	0.176	0.164	0.155	0.148	0.176	12.33	7.7029
56	Pentachloronitrobenzene	0.126	0.116	0.123	0.121	0.120	0.119	0.119	0.121	2.70	7.8304
57	4-Aminobiphenyl	0.681	0.658	0.642	0.632	0.639	0.608	0.613	0.639	3.96	7.8061
58	Pronamide	0.319	0.328	0.338	0.333	0.364	0.330	0.333	0.335	4.19	7.8851
59	Dinoseb	0.157	0.179	0.198	0.202	0.206	0.201	0.206	0.193	9.49	8.0217
60	Disulfoton	0.377	0.317	0.305	0.304	0.300	0.292	0.292	0.312	9.55	8.0231
61	Chrysene-d12	1	1	1	1	1	1	1	1	0	10.8113
62	Methyl parathion	0.153	0.160	0.175	0.170	0.166	0.167	0.160	0.164	4.35	8.4320
63	Ethyl parathion	0.105	0.115	0.128	0.126	0.124	0.131	0.125	0.122	7.52	8.8524
64	4-Nitroquinoline-N-oxide	0.044	0.048	0.057	0.057	0.058	0.059	0.057	0.054	10.89	8.8759
65	Methapyrilene	0.318	0.317	0.296	0.277	0.265	0.253	0.239	0.281	11.07	8.9719
66	Isodrin	0.122	0.110	0.126	0.124	0.116	0.128	0.120	0.121	5.04	9.1741
67	Terphenyl-d14	0.754	0.750	0.786	0.795	0.798	0.820	0.818	0.789	3.53	9.7271
68	Aramite	0.049	0.055	0.063	0.061	0.061	0.060	0.059	0.058	8.11	9.7813
69	p-Dimethylaminoazobenzene	0.200	0.220	0.234	0.226	0.231	0.236	0.233	0.226	5.52	9.8725
70	Chlorobenzilate	0.273	0.274	0.294	0.282	0.284	0.297	0.291	0.285	3.32	9.9216
71	Famphur	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
72	3,3-Dimethylbenzidine	0.241	0.226	0.224	0.215	0.211	0.205	0.201	0.218	6.31	10.2143
73	Kepone	-----	-----	-----	-----	-----	-----	-----	0.000	0.00	0.0000
74	Perylene-d12	1	1	1	1	1	1	1	1	0	12.2513
75	2-Acetylaminofluorene	0.386	0.434	0.462	0.472	0.472	0.467	0.468	0.452	7.07	10.4753
76	7,12-Dimethylben(a)anthracene	0.446	0.461	0.491	0.493	0.492	0.486	0.492	0.480	3.93	11.8314
77	3-Methylcholanthrene	0.477	0.500	0.516	0.524	0.522	0.507	0.525	0.510	3.38	12.5691
78	Dibenzo(a,j)acridine	0.765	0.794	0.836	0.844	0.845	0.838	0.855	0.825	3.99	13.3122

Ave\_%RSD : 5                      Max\_%RSD : 13.8

**Figure 3C: TYPICAL ICAL SUMMARY OF ADDITIONAL ANALYTES APPENDIX IX**

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Instrument ID :E4  
 Beginning DateTime :02/18/14 19:31  
 Spike Units :PPM  
 IC File :RBJ088

Column Spec :Rxi-5Si1MS ID :0.18MM  
 Ending DateTime :02/18/14 21:28  
 HPCHEM Method :SVE4B18C

IDX	Parameters	20	40	50	80	100	120	140	Av_RRF	%_RSD	Av_Rt_M
		21:28 RBJ090	21:08 RBJ089	20:49 RBJ088	20:30 RBJ087	20:10 RBJ086	19:51 RBJ085	19:31 RBJ084			
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	0	3.0246
2	Naphthalene-d8	1	1	1	1	1	1	1	1	0	4.2506
3	Acenaphthene-d10	1	1	1	1	1	1	1	1	0	6.2736
4	Phenanthrene-d10	1	1	1	1	1	1	1	1	0	8.0130
5	Chrysene-d12	1	1	1	1	1	1	1	1	0	10.8083
6	Famphur	0.496	0.484	0.498	0.522	0.523	0.531	0.526	0.511	3.55	10.1741
7	Kepone	0.096	0.094	0.101	0.105	0.104	0.104	0.106	0.101	4.57	10.2749
8	Perylene-d12	1	1	1	1	1	1	1	1	0	12.2447
9	Hexachlorophene	0.042	0.060	0.069	0.084	0.083	0.085	0.088	0.073	23.28	12.0069

Ave\_%RSD : 10.5                      Max\_%RSD : 23.3

Use Least Square Linear Regression with weighting factor of inverse concentration  
 Resp\_Ratio = x0 + x1 \* Amt\_Ratio

IDX	Parameter	x0	x1	CCF
9	Hexachlorophene	-0.02952	0.09531	0.9991

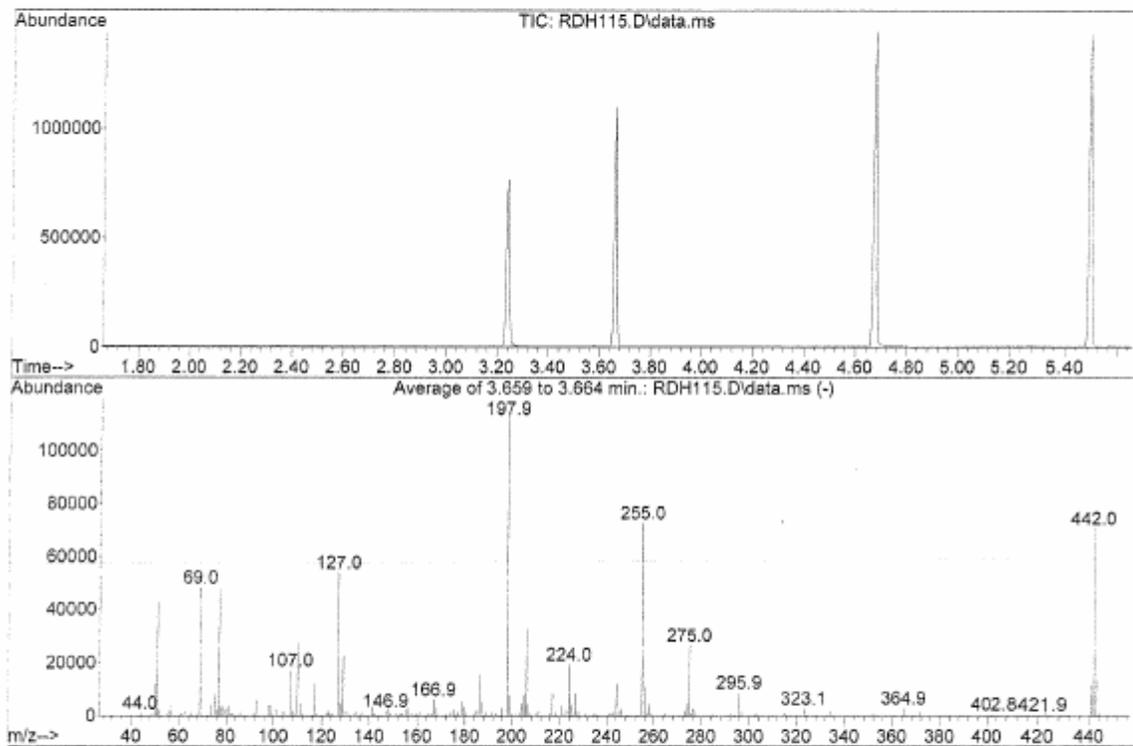
**Figure 4: TYPICAL DFTPP TUNE SUMMARY FOR 8270C**

DFTPP

Data Path : D:\DATA\14D10\  
 Data File : RDH115.D  
 Acq On : 10 Apr 2014 15:20  
 Operator : KV  
 Sample : DFTE7D1001  
 Misc :  
 ALS Vial : 2 Sample Multiplier: 1

Integration File: rteint.p

Method : C:\msdchem\1\METHODS\DFTPP.M  
 Title : DFTPP  
 Last Update : Thu Apr 10 18:47:54 2014



AutoFind: Scans 736, 737, 738; Background Corrected with Scan 728

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	30	60	37.6	42960	PASS
68	69	0.00	2	0.9	440	PASS
69	198	0.00	100	42.3	48224	PASS
70	69	0.00	2	0.0	0	PASS
127	198	40	60	47.1	53784	PASS
197	198	0.00	1	0.0	0	PASS
198	198	100	100	100.0	114120	PASS
199	198	5	9	6.7	7602	PASS
275	198	10	30	23.1	26336	PASS
365	198	1	100	2.5	2845	PASS
441	443	0.01	100	84.1	11129	PASS
442	198	40	100	61.7	70408	PASS
443	442	17	23	18.8	13237	PASS



**Figure 6: TYPICAL CONTINUING CALIBRATION SUMMARY**

CONTINUE\_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7  
 IC\_Beginning DateTime :04/10/14 15:51  
 Spike Amount :25 PPM  
 CC/CV File :RDH129  
 IC File :RDH120

Column Spec :ZB-SemiVoa ID :0.18MM  
 IC\_Ending DateTime :04/10/14 18:06  
 HPChem Method :SVE7D10  
 Date\_Time :04/10/14 20:01

M	IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1		1,4-Dichlorobenzene-d4	40.000	0	236184	1	1	2.866	2.865	0				
2		1,4-Dioxane	24.932	-0.3	69957	0.474	0.475	1.262	1.265	6.36				
3		N-nitrosodimethylamine	22.338	-10.6	87392	0.592	0.663	1.372	1.371	1.92				
4		Pyridine	24.262	-3.0	183749	1.245	1.283	1.396	1.397	2.73				
5		2-Fluorophenol												
6		Phenol-d5												
7		Phenol	22.423	-10.3	197093	1.335	1.489	2.536	2.535	2.55				
8		Aniline	24.673	-1.3	277116	1.877	1.902	2.599	2.598	2.64				
9		bis(2-chloroethyl)ether	23.556	-5.8	148463	1.006	1.067	2.630	2.630	3.66				
10		2-Chlorophenol	22.391	-10.4	169755	1.150	1.284	2.693	2.693	3.29				
11		1,3-Dichlorobenzene	23.135	-7.5	199038	1.348	1.457	2.823	2.822	1.68				
12		1,2-Dichlorobenzene-d4												
13		1,4-Dichlorobenzene	22.931	-8.3	201819	1.367	1.491	2.880	2.879	1.29				
14		Benzyl alcohol	22.739	-9.0	110549	0.749	0.823	2.952	2.956	3.27				
15		1,2-Dichlorobenzene	22.985	-8.1	189990	1.287	1.400	3.010	3.009	2.35				
16		2-Methylphenol	23.651	-5.4	140091	0.949	1.003	3.034	3.032	3.37				
17		bis(2-chloroisopropyl)ether	23.562	-5.8	192513	1.304	1.384	3.077	3.076	3.68				
18		4-Methylphenol	23.276	-6.9	177716	1.204	1.293	3.168	3.168	3.19				
19		N-Nitroso-di-n-propylamine	22.403	-10.4	103672	0.702	0.784	3.192	3.192	3.80				
20		Hexachloroethane	22.794	-8.8	75921	0.514	0.564	3.312	3.313	2.70				
21		Naphthalene-d8	40.000	0	892727	1	1	4.066	4.064	0				
22		Nitrobenzene-d5												
23		Nitrobenzene	22.194	-11.2	183217	0.328	0.370	3.355	3.358	2.02				
24		Isophorone	25.643	2.6	376664	0.675	0.658	3.585	3.587	8.28				
25		2-Nitrophenol	22.051	-11.8	87468	0.157	0.178	3.667	3.664	6.11				
26		2,4-Dimethylphenol	22.620	-9.5	180337	0.323	0.357	3.686	3.687	4.76				
27		Benzoic acid	20.404	-18.4	111108	0.199	0.244	3.767	3.779	10.58				
28		bis(2-Chloroethoxy)methane	23.447	-6.2	190911	0.342	0.365	3.796	3.794	2.74				
29		3,5-Dimethylphenol	21.933	-12.3	180596	0.324	0.369	3.825	3.825	10.56				
30		2,4-Dichlorophenol	22.510	-10.0	168452	0.302	0.335	3.902	3.901	1.95				
31		1,2,4-Trichlorobenzene	22.451	-10.2	185770	0.333	0.371	4.003	4.001	1.93				
32		3,4-Dimethylphenol	23.048	-7.8	224613	0.403	0.437	3.998	4.000	3.88				
33		Naphthalene	22.617	-9.5	514465	0.922	1.019	4.085	4.088	1.20				
34		4-Chloroaniline	23.283	-6.9	216083	0.387	0.416	4.128	4.130	2.49				
35		2,6-Dichlorophenol	23.965	-4.1	151484	0.271	0.283	4.143	4.142	3.59				
36		Hexachlorobutadiene	22.924	-8.3	129760	0.233	0.254	4.220	4.221	2.29				
37		Hydroquinone												
38		4-Chloro-3-methylphenol	21.618	-13.5	134354	0.241	0.278	4.654	4.653	2.25				
39		2-Methylnaphthalene	22.657	-9.4	348859	0.625	0.690	4.846	4.846	1.32				
40		1-Methylnaphthalene	23.384	-6.5	329637	0.591	0.632	4.956	4.958	1.95				
41		Acenaphthene-d10	40.000	0	490143	1	1	6.062	6.061	0				
42		Hexachlorocyclopentadiene	20.470	-18.1	131441	0.429	0.524	5.033	5.032	5.14				
43		2,3,5-Trichlorophenol	28.830	15.3	119762	0.391	0.339	5.033	5.030	1.69				
44		2,4,6-Trichlorophenol	22.427	-10.3	116051	0.379	0.422	5.163	5.160	2.21				
45		2,4,5-Trichlorophenol	24.747	-1.0	130233	0.425	0.429	5.196	5.196	4.44				
46		2-Fluorobiphenyl												
47		2,3,4-Trichlorophenol	24.201	-3.2	109305	0.357	0.369	5.269	5.271	2.79				
48		Biphenyl	24.460	-2.2	445998	1.456	1.488	5.384	5.385	1.35				
49		2-Chloronaphthalene	23.038	-7.8	332776	1.086	1.179	5.404	5.403	1.66				
50		2-Nitroaniline	23.173	-7.3	88791	0.290	0.313	5.514	5.515	4.97				
51		2,6-Dimethylnaphthalene	24.654	-1.4	333443	1.088	1.104	5.571	5.569	1.78				
52		1,4-Dinitrobenzene	24.237	-3.1	50453	0.165	0.170	5.677	5.679	4.29				
53		Dimethylphthalate	23.149	-7.4	452676	1.478	1.596	5.754	5.754	1.32				
54		1,3-Dinitrobenzene	21.607	-13.6	52316	0.171	0.198	5.768	5.767	2.00				
55		2,6-Dinitrotoluene	23.249	-7.0	83399	0.272	0.293	5.812	5.811	1.95				

**Figure 6 (cont.): TYPICAL CONTINUING CALIBRATION SUMMARY**

56	1,2-Dinitrobenzene	24.096	-3.6	33719	0.110	0.114	5.865	5.866	6.89			
57	Acenaphthylene	23.349	-6.6	541096	1.766	1.891	5.889	5.890	0.80			
58	3-Nitroaniline	23.121	-7.5	85538	0.279	0.302	6.000	6.002	2.88			
59	Acenaphthene	22.946	-8.2	349336	1.140	1.242	6.100	6.100	1.87			
60	2,4-Dinitrophenol	22.266	-10.9	43948	0.143	0.161	6.124	6.125	7.03			
61	4-Nitrophenol	22.782	-8.9	68322	0.223	0.245	6.201	6.199	4.70			
62	2,4-Dinitrotoluene	23.512	-6.0	111877	0.365	0.388	6.287	6.287	2.60			
63	Dibenzofuran	22.654	-9.4	458083	1.495	1.650	6.306	6.306	1.34			
64	2,3,5,6-Tetrachlorophenol	23.149	-7.4	83137	0.271	0.293	6.397	6.398	5.24			
65	2,3,4,6-Tetrachlorophenol	20.679	-17.3	98188	0.321	0.387	6.450	6.449	5.82			
66	2,3,5-Trimethylnaphthalene	25.063	0.3	300611	0.981	0.979	6.564	6.563	2.80			
67	Diethylphthalate	23.039	-7.8	402694	1.315	1.426	6.606	6.608	1.28			
68	Fluorene	22.257	-11.0	366505	1.196	1.344	6.719	6.718	1.79			
69	4-Chlorophenyl-phenylether	22.927	-8.3	191484	0.625	0.682	6.733	6.731	0.94			
70	4-Nitroaniline	24.201	-3.2	89054	0.291	0.300	6.733	6.732	2.83			
71	Phenanthrene-d10	40.000	0	919199	1	1	7.796	7.797	0			
72	4,6-Dinitro-2-methylphenol	24.229	-3.1	64140	0.112	0.115	6.775	6.775	14.46			
73	N-Nitrosodiphenylamine	23.461	-6.2	307643	0.535	0.571	6.869	6.870	2.15			
74	Azobenzene	21.761	-13.0	337764	0.588	0.675	6.917	6.918	2.94			
75	2,4,6-Tribromophenol											
76	4-Bromophenyl-phenylether	24.111	-3.6	114341	0.199	0.206	7.301	7.302	1.74			
77	Hexachlorobenzene	22.913	-8.3	118046	0.205	0.224	7.362	7.361	1.26			
78	Pentachlorophenol	23.935	-4.3	81305	0.142	0.148	7.584	7.582	5.21			
79	Dibenzothiophene	23.518	-5.9	511803	0.891	0.947	7.679	7.679	2.00			
80	Phenanthrene	22.694	-9.2	524956	0.914	1.007	7.825	7.822	1.25			
81	Dinoseb											
82	Anthracene	22.872	-8.5	541793	0.943	1.031	7.876	7.877	2.39			
83	Carbazole	23.627	-5.5	511559	0.890	0.942	8.055	8.055	1.57			
84	Di-n-butylphthalate	23.206	-7.2	652551	1.136	1.224	8.470	8.467	4.52			
85	1-Methylphenanthrene	24.442	-2.2	424846	0.740	0.756	8.494	8.493	2.43			
86	Fluoranthene	21.256	-15.0	599853	1.044	1.228	9.098	9.097	7.92			
87	Chrysene-d12	40.000	0	962830	1	1	10.567	10.565	0			
88	Benzidine											
89	Pyrene	22.417	-10.3	635848	1.057	1.178	9.329	9.330	2.15			
90	Terphenyl-d14											
91	Butylbenzylphthalate	24.144	-3.4	287923	0.478	0.495	10.016	10.013	5.78			
92	bis(2-ethylhexyl)adipate	23.931	-4.3	228198	0.379	0.396	10.108	10.109	5.76			
93	3,3'-Dichlorobenzidine	23.151	-7.4	215515	0.358	0.387	10.528	10.528	1.43			
94	Benzo(a)anthracene	22.565	-9.7	612810	1.018	1.128	10.552	10.553	1.48			
95	Chrysene	22.740	-9.0	556264	0.924	1.016	10.591	10.589	2.32			
96	bis(2-Ethylhexyl)phthalate	23.271	-6.9	431382	0.717	0.770	10.601	10.602	4.61			
97	Perylene-d12	40.000	0	949618	1	1	11.926	11.927	0			
98	Di-n-octylphthalate	23.347	-6.6	735924	1.240	1.328	11.217	11.218	6.06			
99	Benzo(b)fluoranthene	22.146	-11.4	572045	0.964	1.088	11.563	11.561	4.73			
100	Benzo(k)fluoranthene	23.377	-6.5	584658	0.985	1.053	11.587	11.588	4.31			
101	Benzo(e)pyrene	24.842	-0.6	591183	0.996	1.002	11.819	11.818	3.35			
102	Benzo(a)pyrene	22.403	-10.4	538072	0.907	1.012	11.868	11.870	4.47			
103	Perylene	22.655	-9.4	557932	0.940	1.037	11.950	11.952	3.09			
104	Indeno(1,2,3-cd)pyrene	23.046	-7.8	631945	1.065	1.155	13.116	13.120	2.77			
105	Dibenzo(a,h)anthracene	23.005	-8.0	513056	0.864	0.939	13.136	13.140	3.75			
106	Benzo(g,h,i)perylene	22.976	-8.1	532510	0.897	0.976	13.470	13.473	2.54			

**Figure 6A: TYPICAL CONTINUING CALIBRATION SUMMARY OF BENZIDINE**

CONTINUE\_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7  
 IC\_Beginning DateTime :04/10/14 20:20  
 Spike Amount :25 PPM  
 CC/CV File :RDH137  
 IC File :RDH134

Column Spec :ZB-SemiVoa ID :0.18MM  
 IC\_Ending DateTime :04/10/14 22:16  
 HPChem Method :SVE7D10A  
 Date\_Time :04/10/14 22:36

M_IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	346380	1	1	2.861	2.862	0				
2	Benzaldehyde												
3	Caprolactam												
4	Phenanthrene-d10	40.000	0	834502	1	1	7.797	7.797	0				
5	Benzidine	23.091	-7.6	372153	0.714	0.773	9.238	9.242	5.16				



**Figure 6B (cont.): TYPICAL CONTINUING CALIBRATION SUMMARY OF ANALYTES APPENDIX IX**

41	Pentachlorobenzene	26.133	4.5	165684	0.561	0.537	6.258	6.256	1.70
42	1-Naphthylamine	26.797	7.2	340129	1.152	1.075	6.393	6.392	1.82
43	2,3,4,6-Tetrachlorophenol								
44	2-Naphthylamine	25.795	3.2	362974	1.230	1.192	6.489	6.488	4.73
45	Thionazin	26.482	5.9	53904	0.183	0.172	6.700	6.699	4.45
46	5-Nitro-o-toluidine	26.914	7.7	98851	0.335	0.311	6.723	6.725	2.86
47	Phenanthrene-d10	40.000	0	897257	1	1	7.796	7.799	0
48	2,4,6-Tribromophenol								
49	Sulfotepp	26.272	5.1	39561	0.071	0.067	7.083	7.084	5.08
50	1,3,5-Trinitrobenzene	26.235	4.9	31338	0.056	0.053	7.163	7.166	12.28
51	Phorate	27.525	10.1	233279	0.416	0.378	7.230	7.230	1.57
52	Phenacetin	26.331	5.3	201737	0.360	0.342	7.239	7.240	2.59
53	Diallate	26.990	8.0	17045	0.030	0.028	7.320	7.322	3.49
54	Dimethoate	26.661	6.6	105997	0.189	0.177	7.409	7.411	5.69
55	Atrazine	26.216	4.9	104341	0.186	0.177	7.504	7.503	7.44
56	Pentachloronitrobenzene	26.988	8.0	66443	0.118	0.110	7.603	7.604	3.80
57	4-Aminobiphenyl	25.698	2.8	383831	0.684	0.666	7.594	7.594	1.11
58	Pronamide	25.511	2.0	193073	0.344	0.337	7.683	7.681	1.71
59	Dinoseb	27.411	9.6	90062	0.161	0.146	7.806	7.806	8.34
60	Disulfoton	26.641	6.6	152036	0.271	0.254	7.825	7.824	4.54
61	Chrysene-d12	40.000	0	912248	1	1	10.562	10.562	0
62	Methyl parathion	27.196	8.8	112386	0.197	0.181	8.224	8.224	4.04
63	Ethyl parathion	27.100	8.4	78996	0.139	0.128	8.649	8.647	6.80
64	4-Nitroquinoline-N-oxide	27.734	10.9	54674	0.096	0.086	8.653	8.653	6.78
65	Methapyrilene	27.737	10.9	185847	0.326	0.294	8.765	8.763	6.69
66	Isodrin	26.753	7.0	73147	0.128	0.120	8.948	8.947	4.26
67	Terphenyl-d14								
68	Aramite	26.566	6.3	31743	0.056	0.052	9.576	9.576	5.90
69	p-Dimethylaminoazobenzene	26.486	5.9	149654	0.262	0.248	9.649	9.650	3.61
70	Chlorobenzilate	27.780	11.1	163928	0.288	0.259	9.703	9.704	5.49
71	3,3-Dimethylbenzidine	26.573	6.3	328992	0.577	0.543	9.986	9.988	7.37
72	Perylene-d12	40.000	0	911888	1	1	11.926	11.926	0
73	2-Acetylaminofluorene	27.124	8.5	273538	0.480	0.442	10.240	10.240	6.11
74	7,12-Dimethylben(a)anthracene	26.412	5.6	262003	0.460	0.435	11.553	11.553	4.04
75	3-Methylcholanthrene	25.078	0.3	274943	0.482	0.481	12.217	12.219	3.80
76	Dibenzo(a,j)acridine	26.759	7.0	450787	0.791	0.739	12.888	12.886	5.22

**Figure 6C: TYPICAL CONTINUING CALIBRATION SUMMARY OF  
 ADDITIONAL ANALYTES APPENDIX IX**

CONTINUE\_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7  
 IC\_Beginning DateTime :04/11/14 14:56  
 Spike Amount :50 PPM  
 CC/CV File :RDH155  
 IC File :RDH152

Column Spec :ZB-SemiVoa ID :0.18MM  
 IC\_Ending DateTime :04/11/14 16:51  
 HPChem Method :SVE7D11C  
 Date\_Time :04/11/14 17:10

M_IDX	Parameters	CC_Con	CC%_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	268288	1	1	2.861	2.862	0				
2	Naphthalene-d8	40.000	0	966151	1	1	4.061	4.063	0				
3	Acenaphthene-d10	40.000	0	521650	1	1	6.062	6.059	0				
4	Phenanthrene-d10	40.000	0	1036706	1	1	7.797	7.796	0				
5	Chrysene-d12	40.000	0	1071961	1	1	10.562	10.567	0				
6	Famphur	51.359	2.7	731295	0.546	0.531	9.952	9.959	2.38				
7	Kepone	48.840	-2.3	136412	0.102	0.104	10.040	10.040	1.74				
8	Perylene-d12	40.000	0	1015463	1	1	11.926	11.933	0				
9	Hexachlorophene	55.518	11.0	158347	0.125	0.111	11.727	11.731	17.61	-0.0344	0.1372		0.9982





**Figure 8: TYPICAL SAMPLE RESULT**

METHOD SW3550B/8270C  
 SEMI VOLATILE ORGANICS BY GC/MS

Client : XYZ, INC.	Date Collected: 06/09/14
Project : CLEAN PROJECT	Date Received: 06/10/14
Batch No. : 14F050	Date Extracted: 06/19/14 12:14
Sample ID: 507-DR-SO-011	Date Analyzed: 06/19/14 21:17
Lab Samp ID: F050-12	Dilution Factor: 1
Lab File ID: RFH297	Matrix : SOIL
Ext Btch ID: SVF031S	% Moisture : 21.5
Calib. Ref.: RDH120	Instrument ID : T-OE7

PARAMETERS	RESULTS (ug/kg)	LOQ (ug/kg)	LOD (ug/kg)
1,2,4-TRICHLOROBENZENE	ND	420	210
1,2-DICHLOROBENZENE	ND	420	210
1,2-DIPHENYLHYDRAZINE	ND	420	210
1,3-DICHLOROBENZENE	ND	420	210
1,4-DICHLOROBENZENE	ND	420	210
2,4,5-TRICHLOROPHENOL	ND	420	210
2,4,6-TRICHLOROPHENOL	ND	420	210
2,4-DICHLOROPHENOL	ND	420	210
2,4-DIMETHYLPHENOL	ND	420	210
2,4-DINITROPHENOL	ND	850	210
2,4-DINITROTOLUENE	ND	420	210
2,6-DINITROTOLUENE	ND	420	210
2-CHLORONAPHTHALENE	ND	420	210
2-CHLOROPHENOL	ND	420	210
2-METHYLNAPHTHALENE	ND	420	210
2-METHYLPHENOL	ND	420	210
2-NITROANILINE	ND	420	210
2-NITROPHENOL	ND	420	210
3,3'-DICHLOROBENZIDINE	ND	420	210
4-METHYLPHENOL (1)	ND	420	210
3-NITROANILINE	ND	420	210
4,6-DINITRO-2-METHYLPHENOL	ND	850	210
4-BROMOPHENYL-PHENYL ETHER	ND	420	210
4-CHLORO-3-METHYLPHENOL	ND	420	210
4-CHLOROANILINE	ND	420	210
4-CHLOROPHENYL-PHENYL ETHER	ND	420	210
4-NITROANILINE	ND	420	210
4-NITROPHENOL	ND	850	210
ACENAPHTHENE	ND	420	210
ACENAPHTHYLENE	ND	420	210
ANTHRACENE	ND	420	210
BENZO(A)ANTHRACENE	ND	420	210
BENZO(A)PYRENE	ND	420	210
BENZO(B)FLUORANTHENE	ND	420	210
BENZO(G,H,I)PERYLENE	ND	420	210
BENZO(K)FLUORANTHENE	ND	420	210
BENZOIC ACID	ND	1700	850
BENZYL ALCOHOL	ND	420	210
2,2'-OXYBIS(1-CHLOROPROPANE)	ND	420	210
BIS(2-CHLOROETHOXY)METHANE	ND	420	210
BIS(2-CHLOROETHYL)ETHER	ND	420	210
BIS(2-ETHYLHEXYL)PHTHALATE	ND	420	210
BUTYLBENZYL PHTHALATE	ND	420	210
CARBAZOLE	ND	420	210
CHRYSENE	ND	420	210
DIBENZO(A,H)ANTHRACENE	ND	420	210
DIBENZOFURAN	ND	420	210
DIETHYL PHTHALATE	ND	420	210
DIMETHYL PHTHALATE	ND	420	210
DI-N-BUTYL PHTHALATE	ND	420	210
DI-N-OCTYL PHTHALATE	ND	420	210
FLUORANTHENE	ND	420	210
FLUORENE	ND	420	210
HEXACHLOROBENZENE	ND	420	210
HEXACHLORO BUTADIENE	ND	420	210
HEXACHLOROETHANE	ND	420	210

**Figure 8 (cont.):** **TYPICAL SAMPLE RESULT**

INDENO(1,2,3-CD)PYRENE	ND	420		210
ISOPHORONE	ND	420		210
NAPHTHALENE	ND	420		210
NITROBENZENE	ND	420		210
N-NITROSODIMETHYLAMINE	ND	420		210
N-NITROSO-DI-N-PROPYLAMINE	ND	420		210
N-NITROSODIPHENYLAMINE (2)	ND	420		210
PENTACHLOROPHENOL	ND	850		210
PHENANTHRENE	ND	420		210
PHENOL	ND	420		210
PYRENE	ND	420		210
BENZIDINE	ND	2500		1100
2,6-DICHLOROPHENOL	ND	420		210
SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
2,4,6-TRIBROMOPHENOL	1950	2548	76.6	35-125
2-FLUOROBIPHENYL	520	849.3	61.2	45-105
2-FLUOROPHENOL	1380	2548	54.1	35-105
NITROBENZENE-D5	455	849.3	53.6	35-100
PHENOL-D5	1460	2548	57.1	40-100
TERPHENYL-D14	610	849.3	71.8	30-125

(1): Cannot be separated from 3-Methylphenol  
 (2): Cannot be separated from Diphenylamine

Figure 9:

## TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA  
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
PROJECT: CLEAN PROJECT  
BATCH NO.: 14F050  
METHOD: SW3550B/8270C

MATRIX: SOIL  
DILUTION FACTOR: 1 1 % MOISTURE: NA  
SAMPLE ID: MBLK15  
LAB SAMP ID: SVF0315B SVF0315L SVF0315C  
LAB FILE ID: RFH277 RFH278 RFH279  
DATE EXTRACTED: 06/19/1412:14 06/19/1412:14 06/19/1412:14 DATE COLLECTED: NA  
DATE ANALYZED: 06/19/1414:50 06/19/1415:09 06/19/1415:28 DATE RECEIVED: 06/19/14  
PREP. BATCH: SVF0315 SVF0315 SVF0315  
CALIB. REF: RDH120 RDH120 RDH120

## ACCESSION:

PARAMETER	BLNK RSLT (ug/kg)	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
1,2,4-Trichlorobenzene	ND	1330	884	66	1330	869	65	2	45-110	50
1,2-Dichlorobenzene	ND	1330	971	73	1330	974	73	0	45-100	50
1,2-Diphenylhydrazine	ND	1330	1070	80	1330	1010	76	6	30-130	50
1,3-Dichlorobenzene	ND	1330	960	72	1330	943	71	2	40-100	50
1,4-Dichlorobenzene	ND	1330	980	74	1330	965	72	2	35-105	50
2,4,5-Trichloropheno1	ND	1330	981	74	1330	939	70	4	50-110	50
2,4,6-Trichloropheno1	ND	1330	1130	85	1330	1080	81	5	45-110	50
2,4-Dichloropheno1	ND	1330	1110	83	1330	1060	80	4	45-110	50
2,4-Dimethylpheno1	ND	1330	1120	84	1330	1080	81	4	30-105	50
2,4-Dinitrophenol	ND	1330	936	70	1330	916	69	2	15-130	50
2,4-Dinitrotoluene	ND	1330	1080	81	1330	1060	80	1	50-115	50
2,6-Dinitrotoluene	ND	1330	1040	78	1330	1030	77	2	50-110	50
2-Chloronaphthalene	ND	1330	1030	77	1330	994	75	4	45-105	50
2-Chloropheno1	ND	1330	1210	91	1330	1170	88	4	45-105	50
2-Methylnaphthalene	ND	1330	942	71	1330	932	70	1	45-105	50
2-Methylpheno1	ND	1330	973	73	1330	956	72	2	40-105	50
2-Nitroaniline	ND	1330	1050	78	1330	1020	76	3	45-120	50
2-Nitrophenol	ND	1330	1280	96	1330	1110	84	14	40-110	50
3,3'-Dichlorobenzidine	ND	1330	1130	84	1330	1060	79	6	10-130	50
4-Methylpheno1	ND	1330	1040	78	1330	985	74	5	40-105	50
3-Nitroaniline	ND	1330	1060	80	1330	1010	76	5	25-110	50
4,6-Dinitro-2-Methylpheno1	ND	1330	1330	100	1330	1280	96	4	30-135	50
4-Bromophenyl-phenyl ether	ND	1330	1170	88	1330	1110	83	6	45-115	50
4-Chloro-3-Methylpheno1	ND	1330	1220	91	1330	1150	86	5	45-115	50
4-Chloroaniline	ND	1330	958	72	1330	936	70	2	10-100	50
4-Chlorophenyl-phenyl ether	ND	1330	1050	79	1330	1010	76	4	45-110	50
4-Nitroaniline	ND	1330	952	71	1330	925	69	3	35-115	50
4-Nitrophenol	ND	1330	865	65	1330	837	63	3	15-140	50
Acenaphthene	ND	1330	1050	79	1330	1030	77	2	45-110	50
Acenaphthylene	ND	1330	1050	79	1330	1030	77	2	45-105	50
Anthracene	ND	1330	1120	84	1330	1060	80	5	55-105	50
Benzo(a)anthracene	ND	1330	1100	82	1330	1060	79	4	50-110	50
Benzo(a)pyrene	ND	1330	1130	84	1330	1080	81	4	50-110	50
Benzo(b)fluoranthene	ND	1330	1080	81	1330	1080	81	1	45-115	50
Benzo(g,h,i)perylene	ND	1330	1130	84	1330	1080	81	4	40-125	50
Benzo(k)fluoranthene	ND	1330	1150	86	1330	1050	79	9	45-125	50
Benzoic Acid	ND	2670	1720	64	2670	1730	65	1	0-110	50
Benzyl Alcohol	ND	1330	963	72	1330	930	70	4	20-125	50
2,2'-oxybis(1-chloropropane)	ND	1330	1120	84	1330	1100	82	2	20-115	50
bis(2-Chloroethoxy)methane	ND	1330	1050	79	1330	1010	76	3	45-110	50
bis(2-Chloroethyl)ether	ND	1330	1020	77	1330	978	73	4	40-105	50
bis(2-Ethylhexyl)phthalate	ND	1330	1200	90	1330	1130	85	6	45-125	50
Butylbenzylphthalate	ND	1330	1220	91	1330	1160	87	5	50-125	50
Carbazole	ND	1330	1040	78	1330	1000	75	4	45-115	50
Chrysene	ND	1330	1090	82	1330	1050	79	4	55-110	50
Dibenzo(a,h)anthracene	ND	1330	1190	89	1330	1150	86	3	40-125	50
Dibenzofuran	ND	1330	1100	82	1330	1060	79	4	50-105	50
Diethylphthalate	ND	1330	1110	83	1330	1050	79	6	50-115	50
Dimethylphthalate	ND	1330	940	71	1330	899	67	4	50-110	50
Di-n-butylphthalate	ND	1330	1290	97	1330	1290	97	0	55-110	50
Di-n-octylphthalate	ND	1330	1150	86	1330	1100	83	4	40-130	50
Fluoranthene	ND	1330	1010	76	1330	966	72	5	55-115	50
Fluorene	ND	1330	1050	79	1330	1010	75	4	50-110	50

**Figure 9: TYPICAL LCS/LCSD SUMMARY**

Hexachlorobenzene	ND	1330	1220	91	1330	1200	90	2	45-120	50
Hexachlorobutadiene	ND	1330	829	62	1330	817	61	2	40-115	50
Hexachloroethane	ND	1330	967	73	1330	966	72	0	35-110	50
Indeno(1,2,3-cd)pyrene	ND	1330	1120	84	1330	1090	82	3	40-120	50
Isophorone	ND	1330	1120	84	1330	1090	82	3	45-110	50
Naphthalene	ND	1330	932	70	1330	907	68	3	40-105	50
Nitrobenzene	ND	1330	964	72	1330	951	71	1	40-115	50
N-Nitrosodimethylamine	ND	1330	1050	78	1330	1040	78	0	20-115	50
n-Nitroso-di-n-propylamine	ND	1330	1070	80	1330	1050	79	2	40-115	50
n-Nitrosodiphenylamine	ND	1330	1220	91	1330	1160	87	5	50-115	50
Pentachlorophenol	ND	1330	1010	75	1330	975	73	3	25-120	50
Phenanthrene	ND	1330	1150	86	1330	1080	81	6	50-110	50
Phenol	ND	1330	1120	84	1330	1070	80	4	40-100	50
Pyrene	ND	1330	1120	84	1330	1050	79	6	45-125	50
Benzidine	ND	2670	1450	54	2670	1390	52	4	30-150	50
2,6-Dichlorophenol	ND	1330	1020	77	1330	1010	76	1	10-150	50

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	QC LIMIT ( % )
2,4,6-Tribromophenol	2000	2110	105	2000	2030	101	35-125
2-Fluorobiphenyl	667	543	81	667	545	82	45-105
2-Fluorophenol	2000	1640	82	2000	1620	81	35-105
Nitrobenzene-d5	667	532	80	667	534	80	35-100
Phenol-d5	2000	1740	87	2000	1690	84	40-100
Terphenyl-d14	667	684	103	667	669	100	30-125

Figure 10:

## TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA  
MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
PROJECT: CLEAN PROJECT  
BATCH NO.: 14F050  
METHOD: SW35508/8270C

MATRIX: SOIL % MOISTURE: 21.5  
DILUTION FACTOR: 1 1  
SAMPLE ID: 507-DR-SO-011  
LAB SAMP ID: F050-12 F050-12M F050-12S  
LAB FILE ID: RFH297 RFH282 RFH283  
DATE EXTRACTED: 06/19/1412:14 06/19/1412:14 06/19/1412:14 DATE COLLECTED: 06/09/14  
DATE ANALYZED: 06/19/1421:17 06/19/1416:26 06/19/1416:46 DATE RECEIVED: 06/10/14  
PREP. BATCH: SVF0315 SVF0315 SVF0315  
CALIB. REF: RDH120 RDH120 RDH120

## ACCESSION:

PARAMETER	SPL RSLT (ug/kg)	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
1,2,4-Trichlorobenzene	ND	1700	1120	66	1700	1130	67	1	45-110	50
1,2-Dichlorobenzene	ND	1700	1170	69	1700	1190	70	1	45-100	50
1,2-Diphenylhydrazine	ND	1700	1370	81	1700	1420	84	4	30-130	50
1,3-Dichlorobenzene	ND	1700	1160	69	1700	1180	69	1	40-100	50
1,4-Dichlorobenzene	ND	1700	1180	69	1700	1170	69	1	35-105	50
2,4,5-Trichlorophenol	ND	1700	1340	79	1700	1400	82	4	50-110	50
2,4,6-Trichlorophenol	ND	1700	1510	89	1700	1560	92	3	45-110	50
2,4-Dichlorophenol	ND	1700	1370	81	1700	1430	84	4	45-110	50
2,4-Dimethylphenol	ND	1700	1370	81	1700	1410	83	3	30-105	50
2,4-Dinitrophenol	ND	1700	1330	79	1700	1310	77	2	15-130	50
2,4-Dinitrotoluene	ND	1700	1450	85	1700	1480	87	2	50-115	50
2,6-Dinitrotoluene	ND	1700	1370	80	1700	1430	84	5	50-110	50
2-Chloronaphthalene	ND	1700	1350	79	1700	1400	82	4	45-105	50
2-Chlorophenol	ND	1700	1300	77	1700	1330	78	2	45-105	50
2-Methylnaphthalene	ND	1700	1180	70	1700	1220	72	3	45-105	50
2-Methylphenol	ND	1700	1180	69	1700	1190	70	1	40-105	50
2-Nitroaniline	ND	1700	1410	83	1700	1440	85	2	45-120	50
2-Nitrophenol	ND	1700	1390	82	1700	1440	85	4	40-110	50
3,3'-Dichlorobenzidine	ND	1700	1470	86	1700	1570	92	7	10-130	50
4-Methylphenol	ND	1700	1380	81	1700	1300	76	6	40-105	50
3-Nitroaniline	ND	1700	1400	82	1700	1450	86	4	25-110	50
4,6-Dinitro-2-Methylphenol	ND	1700	1770	104	1700	1850	109	5	30-135	50
4-Bromophenyl-phenyl ether	ND	1700	1500	88	1700	1590	93	6	45-115	50
4-Chloro-3-Methylphenol	ND	1700	1520	89	1700	1540	91	1	45-115	50
4-Chloroaniline	ND	1700	1160	68	1700	1210	71	4	10-100	50
4-Chlorophenyl-phenyl ether	ND	1700	1330	78	1700	1370	81	3	45-110	50
4-Nitroaniline	ND	1700	1190	70	1700	1270	75	6	35-115	50
4-Nitrophenol	ND	1700	1180	69	1700	1220	72	3	15-140	50
Acenaphthene	ND	1700	1370	81	1700	1410	83	3	45-110	50
Acenaphthylene	ND	1700	1370	80	1700	1430	84	4	45-105	50
Anthracene	ND	1700	1390	82	1700	1470	87	6	55-105	50
Benzo(a)anthracene	ND	1700	1360	80	1700	1420	84	5	50-110	50
Benzo(a)pyrene	ND	1700	1390	82	1700	1480	87	7	50-110	50
Benzo(b)fluoranthene	ND	1700	1410	83	1700	1560	92	10	45-115	50
Benzo(g,h,i)perylene	ND	1700	1380	81	1700	1430	84	4	40-125	50
Benzo(k)fluoranthene	ND	1700	1390	82	1700	1380	81	1	45-125	50
Benzoic Acid	ND	3400	15300	45	3400	1870	55	20	0-110	50
Benzyl Alcohol	ND	1700	1180	69	1700	1180	69	0	20-125	50
2,2'-oxybis(1-chloropropane)	ND	1700	1350	79	1700	1360	80	1	20-115	50
bis(2-Chloroethoxy)methane	ND	1700	1260	74	1700	1300	77	4	45-110	50
bis(2-Chloroethyl)ether	ND	1700	1190	70	1700	1230	73	3	40-105	50
bis(2-Ethylhexyl)phthalate	ND	1700	1400	82	1700	1520	89	8	45-125	50
Butylbenzylphthalate	ND	1700	1400	82	1700	1510	89	8	50-125	50
Carbazole	ND	1700	1340	79	1700	1450	85	8	45-115	50
Chrysene	ND	1700	1340	79	1700	1430	84	6	55-110	50
Dibenzo(a,h)anthracene	ND	1700	1420	84	1700	1510	89	6	40-125	50
Dibenzofuran	ND	1700	1420	84	1700	1470	87	3	50-105	50
Diethylphthalate	ND	1700	1390	82	1700	1450	85	4	50-115	50
Dimethylphthalate	ND	1700	1210	71	1700	1240	73	3	50-110	50
Di-n-butylphthalate	ND	1700	1470	87	1700	1620	96	10	55-110	50
Di-n-octylphthalate	ND	1700	1400	83	1700	1470	86	5	40-130	50
Fluoranthene	ND	1700	1380	81	1700	1470	87	6	55-115	50
Fluorene	ND	1700	1340	79	1700	1390	82	3	50-110	50

Figure 10 (cont.):

## TYPICAL MS/MSD SUMMARY

Hexachlorobenzene	ND	1700	1500	89	1700	1600	94	6	45-120	50
Hexachlorobutadiene	ND	1700	1050	62	1700	1050	62	1	40-115	50
Hexachloroethane	ND	1700	1390	82	1700	1180	70	16	35-110	50
Indeno(1,2,3-cd)pyrene	ND	1700	1370	81	1700	1460	86	6	40-120	50
Isophorone	ND	1700	1350	80	1700	1250	74	8	45-110	50
Naphthalene	ND	1700	1160	68	1700	1170	69	1	40-105	50
Nitrobenzene	ND	1700	1190	70	1700	1200	71	1	40-115	50
N-Nitrosodimethylamine	ND	1700	1270	75	1700	1250	74	1	20-115	50
n-Nitroso-di-n-propylamine	ND	1700	1290	76	1700	1320	78	2	40-115	50
n-Nitrosodiphenylamine	ND	1700	1660	97	1700	1720	101	4	50-115	50
Pentachlorophenol	ND	1700	1360	80	1700	1470	87	8	25-120	50
Phenanthrene	ND	1700	1430	84	1700	1530	90	7	50-110	50
Phenol	ND	1700	1360	80	1700	1370	81	1	40-100	50
Pyrene	ND	1700	1240	73	1700	1330	78	7	45-125	50
Benzidine	ND	3400	1260J	37	3400	1210J	36	4	30-150	50
2,6-Dichlorophenol	ND	1700	1250	74	1700	1310	77	4	10-150	50

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	QC LIMIT ( % )
2,4,6-Tribromophenol	2550	2730	107	2550	2930	115	35-125
2-Fluorobiphenyl	849	724	85	849	752	89	45-105
2-Fluorophenol	2550	1930	76	2550	1940	76	35-105
Nitrobenzene-d5	849	659	78	849	668	79	35-100
Phenol-d5	2550	2070	81	2550	2100	82	40-100
Terphenyl-d14	849	831	98	849	808	95	30-125

**Figure 11: TYPICAL CASE NARRATIVE**

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN PROJECT  
SDG : 14F050

METHOD SW3550B/8270C  
SEMI VOLATILE ORGANICS BY GC/MS

A total of nineteen (19) soil samples were received on 06/10/14 for Semivolatile Organics by GC/MS analysis, Method SW3550B/8270C in accordance with Project QAPP, 2/2013.

Holding Time

Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration

Instrument tune check was performed prior to calibration. Instrument mass ratios as well as DDT breakdown were evaluated. Results were within acceptance criteria. Tailing factor for Benzidine and Pentachlorophenol were also verified and results were within the method limits. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms of ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Results were compliant to project requirement.

Lab Control Sample

Two (2) sets of LCS/LCD were analyzed with the samples in this SDG. Percent recoveries for SVF031SL/C were all within QC limits. Percent recoveries for A9F031SL/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the samples in this SDG. Percent recoveries for F050-12M/S were within project QC limits.

Surrogate

Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits. Refer to sample result forms for details.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met; otherwise, anomalies were discussed within the associated QC parameter. Sample F050-14 was initially analyzed at dilution due to dark-colored appearance in extract.

**Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
DFTPP Tune Check	Prior to calibration (ICAL, ICV or CCV)	Refer to Table 7	Re-tune instrument and verify		
Breakdown Check	Prior to calibration (ICAL, ICV or CCV)	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, Benzidine tailing ≤ 3, pentachlorophenol tailing ≤ 5	Clean the injection port and repeat breakdown check. If problem persist cut or replace column.		
Multi point Initial Calibration (ICAL) minimum of 5 points	Initially; as needed	SPCCs (Table 11): Average RF ≥ 0.05 CCCs (Table 10): RSD ≤ 30% 1.) If RRF is applied, then RSD ≤ 15% 2.) If 1st order is applied, then r ≥ 0.995 with min 5 pt ICAL 3.) If 2nd order is applied, then COD ≥ 0.99 with min 6 pt ICAL	Check for outliers. Otherwise, optimize the instrument then repeat initial calibration.		
Initial Calibration Verification (ICV)	After initial calibration	In the absence of PSR All analytes within ± 20% of expected value *Exception Analytes within ± 35% of expected value	Verify second source standard. Prepare fresh standard and re-run ICV. If that fails, optimize instrument and repeat ICAL.		
Evaluation of relative retention times (RRT)	Each sample	Within ± 0.06 RRT units of the RRT	Correct the problem then re-analyze all samples analyzed since the last retention time check.		
Continuing Calibration Verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs: Min. RF same as ICAL CCCs: %Diff ≤ 20% (when using RFs) or drift (when using least square regression or non-linear calibration)	Correct the problem then repeat initial calibration.		
Internal Standard (IS)	All samples	Retention time ±30 seconds from retention time of the mid-point standard in the ICAL.; EICP area within -50% to +100% of ICAL mid-point standard	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning		
Method Blank (MB)	One per preparation batch	In the absence of PSR apply No analytes detected > ½ LOQ	Rule out instrument contamination by re-analyzing the MB. If problem persist refer to PSR. In the absence of PSR, report NDs and results >10X of the MB concentration. Otherwise, cure contamination source, re-prep and re-analyze method blank and all associated samples.		
LCS	One LCS per preparation	In the absence of PSR default to EMAX QC Limits	Re-prep and re-analyze the LCS and all associated samples		
MS/MSD	One MS/MSD per every 20 project samples per matrix	In the absence of PSR default to EMAX QC Limits	Check if sample was properly spiked. If indicative of matrix interference, discuss in case narrative, otherwise re-prep and re-analyze the sample		
Surrogate	Every sample, MB, LCS, MS/MSD, DCC	In the absence of PSR, at least 2 out of 3 Acids and 2 out of 3 BN surrogates are within EMAX QC Limits.	Check if sample was properly spiked. If indicative of matrix interference, discuss in case narrative, otherwise re-prep and re-analyze the sample		
<b>Comments:</b> For flagging criteria refer to PSR. Otherwise, if MB is non-compliant, apply "B" to specific analyte(s) on all associated samples, apply "J" to all values between LOD and LOQ. <b>*Exception Analytes</b> - Analytes known to have erratic chromatographic behavior: Benzidine, 4,6-dinitro-2-methylphenol, 4-chroanaline, benzyl alcohol, n-Nitrosodimethylamine, 4-nitrophenol, 2-nitroaniline, Pyridine, Benzoic Acid, and 3-nitroaniline			Reviewed By:		
			Date:		

**Appendix 2: DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: EPA 8270**

Sample Prep SOP: EMAX-3550 Rev. 4  
Analytical SOP: EMAX-8270 Rev. 5  
Conc Unit: µg/kg  
Sample Amt(g): 15  
Extract Volume (mL): 2

Instrument ID: E7  
Extraction date: 3/22 & 3/24/14  
Extracted by: J. Villena  
Analysis date: 3/24/2014  
Analyzed by: D. Jun

PARAMETER	SVC041SL	SVC041SC	SVC042SL	SVC042SC	TV	Ave. Conc.	Ave. %Rec	SD	QC Criteria	COMMENTS
	RCH420	RCH421	RCH422	RCH423						
Acenaphthene	2218	2120	2086	1995	2667	2105	79	92.0	50 - 130	PASSED
Acenaphthylene	2284	2197	2166	2127	2667	2194	82	66.8	50 - 130	PASSED
Aniline	2315	2090	2115	2086	2667	2152	81	109.5	40 - 130	PASSED
Anthracene	2242	2117	2117	2034	2667	2128	80	86.0	50 - 130	PASSED
Azobenzene	2095	1960	1906	1873	2667	1958	73	97.9	30 - 160	PASSED
Benzidine	1865	1743	1869	1716	2667	1798	67	80.4	20 - 130	PASSED
Benzo(a)anthracene	2620	2534	2556	2506	2667	2554	96	48.7	60 - 130	PASSED
benzo(a)pyrene	2478	2411	2481	2392	2667	2440	92	45.8	50 - 130	PASSED
Benzo(b)fluoranthene	2660	2560	2457	2403	2667	2520	94	113.8	60 - 130	PASSED
Benzo(g,h,i)perylene	2505	2378	2659	2356	2667	2475	93	139.3	50 - 130	PASSED
Benzo(k)fluoranthene	2327	2322	2510	2366	2667	2381	89	88.0	60 - 130	PASSED
Benzoic Acid	1116	1090	1330	1206	2667	1185	44	108.3	20 - 130	PASSED
Benzyl Alcohol	2144	1857	1920	1977	2667	1975	74	123.3	50 - 130	PASSED
bis(2-chloroethoxy)methane	2354	2119	2139	1997	2667	2152	81	148.4	50 - 130	PASSED
bis(2-chloroethyl)ether	2160	2073	2073	2011	2667	2079	78	61.4	50 - 130	PASSED
bis(2-chloroisopropyl)ether	2962	2690	2732	2679	2667	2766	104	132.9	30 - 130	PASSED
bis(2-Ethylhexyl)phthalate	2547	2411	2442	2391	2667	2448	92	69.5	60 - 130	PASSED
4-Bromophenyl-phenylether	2274	2232	2179	2101	2667	2197	82	74.5	50 - 130	PASSED
Butylbenzylphthalate	2715	2587	2617	2524	2667	2611	98	79.4	60 - 130	PASSED
Carbazole	2472	2345	2381	2296	2667	2373	89	74.4	50 - 130	PASSED
4-Chloro-3-methylphenol	2623	2485	2427	2239	2667	2443	92	159.5	50 - 130	PASSED
4-Chloroaniline	2273	2046	2002	1981	2667	2076	78	134.3	40 - 130	PASSED
2-Chloronaphthalene	2181	2071	2016	1972	2667	2060	77	90.5	50 - 130	PASSED
2-Chlorophenol	2202	1982	1992	2007	2667	2046	77	104.4	40 - 130	PASSED
4-Chlorophenyl-phenylether	2357	2305	2263	2185	2667	2278	85	72.7	50 - 130	PASSED
Chrysene	2707	2564	2572	2496	2667	2585	97	88.1	60 - 130	PASSED
Dibenzo(a,h)anthracene	2578	2466	2529	2447	2667	2505	94	60.1	60 - 130	PASSED
Dibenzofuran	2256	2126	2080	2037	2667	2125	80	95.1	50 - 130	PASSED
1,2-Dichlorobenzene	2165	1986	2030	2062	2667	2061	77	76.1	50 - 130	PASSED
1,3-Dichlorobenzene	2224	2011	2130	2076	2667	2110	79	90.4	50 - 130	PASSED
1,4-Dichlorobenzene	2198	1983	2104	2025	2667	2077	78	94.5	40 - 130	PASSED
3,3'-Dichlorobenzidine	2972	2745	2853	2746	2667	2829	106	107.8	60 - 130	PASSED
2,4-Dichlorophenol	2219	2044	2038	1929	2667	2058	77	120.1	40 - 130	PASSED
2,6-Dichlorophenol	2343	2146	2232	2037	2667	2190	82	129.7	30 - 160	PASSED
Diethylphthalate	2338	2210	2217	2111	2667	2219	83	92.6	60 - 130	PASSED
2,4-Dimethylphenol	2259	2028	2021	1972	2667	2070	78	128.7	40 - 130	PASSED
Dimethylphthalate	2804	2661	2565	2458	2667	2622	98	147.0	60 - 130	PASSED
Di-n-butylphthalate	2317	2189	2268	2143	2667	2229	84	78.0	60 - 130	PASSED
4,6-Dinitro-2-methylphenol	2332	2126	2224	2260	2667	2236	84	85.7	50 - 140	PASSED
2,4-Dinitrophenol	2325	2218	2275	2177	2667	2249	84	64.6	20 - 130	PASSED

**Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: EPA 8270**

Sample Prep SOP: EMAX-3550 Rev. 4  
Analytical SOP: EMAX-8270 Rev. 5  
Conc Unit: µg/kg  
Sample Amt(g): 15  
Extract Volume (mL): 2

Instrument ID: E7  
Extraction date: 3/22 & 3/24/14  
Extracted by: J. Villena  
Analysis date: 3/24/2014  
Analyzed by: D. Jun

PARAMETER	SVC0415L	SVC0415C	SVC0425L	SVC0425C	TV	Ave. Conc.	Ave. %Rec	SD	QC Criteria	COMMENTS
	RCH420	RCH421	RCH422	RCH423						
2,4-Dinitrotoluene	2590	2285	2381	2342	2667	2400	90	132.7	60 - 130	PASSED
2,6-Dinitrotoluene	2386	2378	2246	2237	2667	2312	87	81.4	60 - 130	PASSED
Di-n-octylphthalate	2775	2698	2799	2631	2667	2726	102	76.5	60 - 130	PASSED
Fluoranthene	2577	2483	2519	2332	2667	2478	93	104.4	60 - 130	PASSED
Fluorene	2203	2125	2036	2023	2667	2097	79	84.1	50 - 130	PASSED
Hexachlorobenzene	2174	1969	2021	1918	2667	2020	76	110.5	50 - 130	PASSED
Hexachlorobutadiene	2263	2073	2072	2043	2667	2113	79	101.5	40 - 130	PASSED
Hexachlorocyclopentadiene	1704	1651	1573	1575	2667	1626	61	63.8	20 - 130	PASSED
Hexachloroethane	2251	2071	2102	2082	2667	2127	80	84.0	40 - 130	PASSED
Indeno(1,2,3-cd)pyrene	2498	2420	2495	2372	2667	2446	92	61.1	60 - 130	PASSED
Isophorone	2087	1949	1960	1865	2667	1966	74	91.5	50 - 130	PASSED
1-Methylnaphthalene	2389	2178	2176	2101	2667	2211	83	123.9	50 - 130	PASSED
2-Methylnaphthalene	2689	2520	2428	2406	2667	2511	94	128.8	50 - 130	PASSED
2-Methylphenol	2222	1996	2076	1972	2667	2066	77	113.0	50 - 130	PASSED
4-Methylphenol	2298	2289	2267	2066	2667	2230	84	109.9	50 - 130	PASSED
Naphthalene	2208	2000	2030	1977	2667	2054	77	105.0	50 - 130	PASSED
2-Nitroaniline	2616	2515	2414	2705	2667	2563	96	126.0	50 - 130	PASSED
3-Nitroaniline	2503	2434	2364	2253	2667	2388	90	106.6	50 - 130	PASSED
4-Nitroaniline	2657	2497	2560	2487	2667	2550	96	78.3	50 - 130	PASSED
Nitrobenzene	2210	2044	2027	1995	2667	2069	78	96.3	50 - 130	PASSED
2-Nitrophenol	2631	2520	2405	2372	2667	2482	93	117.9	40 - 130	PASSED
4-Nitrophenol	2333	2258	2316	2303	2667	2303	86	32.0	40 - 130	PASSED
n-Nitrosodimethylamine	1914	1721	1784	1684	2667	1776	67	101.1	40 - 130	PASSED
n-Nitroso-di-n-propylamine	2214	1938	1972	1933	2667	2014	76	134.2	50 - 130	PASSED
n-Nitrosodiphenylamine	2324	2202	2208	2221	2667	2239	84	57.4	40 - 130	PASSED
Pentachlorophenol	2233	2091	2121	2013	2667	2114	79	91.5	30 - 130	PASSED
Phenanthrene	2216	2105	2104	2030	2667	2114	79	76.7	50 - 130	PASSED
Phenol	2125	1980	2038	1906	2667	2012	75	92.5	50 - 130	PASSED
Pyrene	2549	2398	2434	2326	2667	2427	91	93.1	50 - 130	PASSED
Pyridine	1679	1552	1668	1591	2667	1623	61	61.2	30 - 130	PASSED
2,3,4,6-Tetrachlorophenol	2374	2214	2222	2137	2667	2237	84	99.4	30 - 160	PASSED
1,2,4-Trichlorobenzene	2199	1997	2070	1968	2667	2059	77	102.8	40 - 130	PASSED
2,4,5-Trichlorophenol	2330	2223	2257	2070	2667	2220	83	109.8	50 - 130	PASSED
2,4,6-Trichlorophenol	2170	2040	1988	1995	2667	2048	77	84.3	50 - 130	PASSED
2-Fluorophenol	3555	3234	3374	3295	4000	3364	84	139.2	40 - 130	PASSED
Phenol-d5	3636	3338	3424	3264	4000	3415	85	161.0	50 - 130	PASSED
2-Fluorobiphenyl	1122	1065	1093	1052	1333	1083	81	31.3	40 - 130	PASSED
Nitrobenzene-d5	1136	1009	1066	974	1333	1046	78	70.7	40 - 130	PASSED
2,4,6-Tribromophenol	3419	3256	3355	3093	4000	3281	82	142.0	50 - 130	PASSED
Terphenyl-d14	1489	1427	1478	1453	1333	1462	110	27.6	60 - 130	PASSED

Appendix 3:

DEMONSTRATION OF CAPABILITY  
FOR APPENDIX IX COMPOUNDS

DEMONSTRATION OF CAPABILITY  
METHOD: SW 3550C / SW 8270C/D  
APPENDIX IX COMPOUNDS

Sample Prep SOP: EMAX-3550  
Analytical SOP: EMAX-8270  
Conc Unit:  $\mu\text{g}/\text{kg}$   
Sample Amt(gm): 30  
Extract Volume (mL): 2

Instrument ID: E4  
Extraction date: 6/24/2011  
Extracted by: J. Villena  
Analysis date: 6/27 & 6/28/11  
Analyzed by: D. Cheung

PARAMETER	RFJ303	RFJ304	RFJ343	RFJ344	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	A9F0445L	A9F0445C	A9F0455L	A9F0455C							
Acetophenone	1225	1231	1632	1204	1600	1323	83	206.5	16	30 - 150	PASSED
2-acetylaminofluorene	1433	1485	1884	1669	1600	1618	101	204.7	13	30 - 150	PASSED
4-Aminobiphenyl	1502	1589	1895	1650	1600	1659	104	168.6	10	30 - 150	PASSED
Aramite	1526	1586	2057	1770	1600	1735	108	238.9	14	30 - 150	PASSED
Atrazine	1508	1500	1858	1643	1600	1627	102	167.2	10	30 - 150	PASSED
Biphenyl	1235	1256	1600	1247	1600	1334	83	177.5	13	30 - 130	PASSED
Chlorobenzilate	1390	1454	1825	1541	1600	1553	97	192.1	12	30 - 150	PASSED
1-Chloronaphthalene	1266	1295	1616	1277	1600	1363	85	168.7	12	30 - 150	PASSED
Diallate	1272	1303	1638	1490	1600	1426	89	171.3	12	30 - 150	PASSED
Dibenzo(a,j)acridine	1272	1319	1660	1385	1600	1409	88	173.5	12	30 - 150	PASSED
2,6-Dichlorophenol	1228	1225	1633	1248	1600	1334	83	200.0	15	30 - 150	PASSED
Dimethoate	1563	1616	2017	1741	1600	1734	108	202.6	12	30 - 150	PASSED
p-Dimethylaminoazobenze	1374	1394	1776	1544	1600	1522	95	185.8	12	30 - 150	PASSED
7,12-Dimethylben(a)anthracene	1383	1428	1764	1547	1600	1530	96	170.5	11	30 - 150	PASSED
3,3-Dimethylbenzidine	1383	1427	988	1516	1600	1329	83	233.7	18	10 - 150	PASSED
3,4-Dimethylphenol	1172	1194	1610	1291	1600	1317	82	202.2	15	30 - 150	PASSED
Dinoseb	1324	1419	1836	1556	1600	1534	96	222.8	15	30 - 150	PASSED
1,4-Dioxane	1097	1122	1376	1041	1600	1159	72	148.7	13	30 - 150	PASSED
Diphenyl ether	1219	1237	1565	1243	1600	1316	82	166.2	13	30 - 150	PASSED
Disulfoton	1356	1414	1782	1554	1600	1526	95	189.7	12	30 - 150	PASSED
Ethyl methacrylate	1121	1170	1440	1082	1600	1203	75	162.0	13	30 - 150	PASSED
Ethyl methanesulfonate	1217	1252	1589	1202	1600	1315	82	184.0	14	30 - 150	PASSED
Ethyl parathion	1364	1445	1880	1525	1600	1554	97	227.6	15	30 - 150	PASSED
Hexachloropropene	1226	1261	1578	1201	1600	1317	82	176.0	13	30 - 150	PASSED
Isodrin	1308	1276	1642	1449	1600	1419	89	166.5	12	30 - 150	PASSED
Isosafrole	1427	1445	1867	1473	1600	1553	97	210.5	14	30 - 150	PASSED
3-Methylcholanthrene	1407	1445	1811	1579	1600	1561	98	182.6	12	30 - 150	PASSED
Methyl methanesulfonate	1219	1216	1423	1154	1600	1253	78	117.3	9	30 - 150	PASSED
Methyl parathion	1418	1509	1923	1667	1600	1630	102	221.1	14	30 - 150	PASSED
1,4-Naphthoquinone	1337	1376	1702	1517	1600	1483	93	165.3	11	10 - 150	PASSED
1-Naphthylamine	1285	1288	1660	1396	1600	1407	88	176.1	13	30 - 150	PASSED
2-Naphthylamine	1425	1418	1806	1586	1600	1559	97	182.3	12	30 - 150	PASSED
N-nitrosodiethylamine	1207	1253	1604	1202	1600	1317	82	192.9	15	30 - 150	PASSED
N-Nitrosomethylethylamine	1187	1252	1577	1171	1600	1297	81	190.0	15	30 - 150	PASSED
N-Nitrosomorpholine	1206	1249	1627	1210	1600	1323	83	203.4	15	30 - 150	PASSED
N-Nitrosodi-n-butylamine	1263	1295	1764	1399	1600	1430	89	230.0	16	30 - 150	PASSED
N-Nitrosopiperidine	1231	1247	1647	1266	1600	1348	84	200.2	15	30 - 150	PASSED
N-Nitrosopyrrolidine	1194	1242	1678	1267	1600	1345	84	224.0	17	30 - 150	PASSED
5-Nitro-o-toluidine	1425	1465	1829	1506	1600	1556	97	185.0	12	30 - 150	PASSED
4-Nitroquinoline-N-oxide	1402	1493	1737	1567	1600	1550	97	142.1	9	10 - 150	PASSED
Pentachlorobenzene	1257	1306	1656	1355	1600	1393	87	179.7	13	30 - 150	PASSED

Appendix 3 (cont.):

DEMONSTRATION OF CAPABILITY  
FOR APPENDIX IX COMPOUNDS

DEMONSTRATION OF CAPABILITY  
METHOD: SW 3550C / SW 8270C/D  
APPENDIX IX COMPOUNDS

Sample Prep SOP: EMAX-3550  
Analytical SOP: EMAX-8270  
Conc Unit: µg/Kg  
Sample Amt(gm): 30  
Extract Volume (mL): 2

Instrument ID: E4  
Extraction date: 6/24/2011  
Extracted by: J. Villena  
Analysis date: 6/27 & 6/28/11  
Analyzed by: D. Cheung

PARAMETER	RFJ303	RFJ304	RFJ343	RFJ344	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	A9F0445L	A9F0445C	A9F0455L	A9F0455C							
Pentachloroethane	1203	1195	1487	1106	1600	1248	78	165.2	13	30 - 150	PASSED
Pentachloronitrobenzene	1354	1424	1721	1553	1600	1513	95	161.4	11	30 - 150	PASSED
Phenacetin	1476	1527	1853	1585	1600	1610	101	167.8	10	30 - 150	PASSED
p-phenylenediamine	826	886	1211	844	1600	942	59	180.9	19	10 - 150	PASSED
Phorate	1340	1377	1752	1524	1600	1498	94	186.8	12	30 - 150	PASSED
2-Picoline	1036	1063	1338	956	1600	1098	69	165.9	15	10 - 150	PASSED
pronamide	1456	1506	1877	1625	1600	1616	101	187.7	12	30 - 150	PASSED
Safrole	1221	1263	1667	1277	1600	1357	85	208.0	15	30 - 150	PASSED
Sulfotepp	1317	1376	1723	1534	1600	1487	93	181.8	12	30 - 150	PASSED
1,2,4,5-Tetrachlorobenzene	1233	1269	1624	1246	1600	1343	84	187.8	14	30 - 150	PASSED
Thionazin	1364	1365	1809	1540	1600	1520	95	210.1	14	30 - 150	PASSED
o-toluidine	1387	1401	1769	1345	1600	1475	92	196.9	13	30 - 150	PASSED
O,O,O-triethyl phosphorothi	1218	1230	1612	1237	1600	1324	83	192.2	15	30 - 150	PASSED
1,3,5-Trinitrobenzene	1432	1566	1952	1464	1600	1604	100	239.1	15	10 - 150	PASSED
2-Fluorophenol	1602	1611	1767	1545	2000	1632	82	95.0	6	40 - 130	PASSED
Pheno-d5	1584	1616	1876	1623	2000	1675	84	135.3	8	50 - 130	PASSED
Nitrobenzene-d5	522	519	605	531	667	544	82	40.5	7	40 - 130	PASSED
2-Fluorobiphenyl	527	534	617	545	667	556	83	41.2	7	50 - 130	PASSED
2,4,6-Tribromophenol	1788	1860	2086	2059	2000	1948	97	147.2	8	50 - 130	PASSED
Terphenyl-d14	658	678	764	742	667	710	106	50.3	7	50 - 130	PASSED





8270FA:

ANALYTICAL RUN LOG



ANALYSIS LOG FOR SEMIVOLATILES

SOP  EMAX-8270 Rev. No. \_  EMAX-8270D Rev. No. \_  EMAX-8270SIM Rev. No. \_  EMAX-CLPSVOA  EMAX-M8270SIM Rev. No. \_  EMAX-625 Rev. No. \_

Book #: AF0-002

Method File:	Tune File:	Start Date/Time:	End Date/Time:	Matrix		Notes	Instrument No:		F0
				S	W				
ANALYTICAL BATCH : _____	Preparative Batch	Data File Name	Run ID	DF			INITIAL CALIBRATION REFERENCE		
							Date		
							ICAL ID		
							Standards		
							Name	ID	Conc. (mg/L)
							DFTPP		
							INT. STD.		
							ICV		
							DCC		
							BENZIDINE		
							APP 9		
							APP 9 ADD		
							Solvent	ID	
							CH <sub>2</sub> Cl <sub>2</sub>		
							DATA FILE		
							Electronic Data Archival		
							Location	Date	
							HPCHEM_SVOA/TOFO		
							Micropipette ID:	<input type="checkbox"/> PO97A-02 <input type="checkbox"/> PO97A-03 <input type="checkbox"/> PO00-01	
						Comments:	_____		
						Analyzed By:	_____		
						Date Disposed:	_____		
						Disposed By:	_____		
						This page is checked during data review.			



SOP REVIEW FORM

EMAX-8270SIM

Rev. 2-

SEMIVOLATIVE ORGANICS BY GC/MS

SOP No.

Revision Number

Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	/			
Summary of Method	/			
Detection Limits	/			
Dynamic Range	/			
Sample Holding Time & Preservation	/			
Associated SOPs	/			
Safety	/			I have read all MSDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents	/			
Standards	/			
Procedures	/			
- Sample Preparation	/			
- Instrument Parameters	/			
- Calibration	/			
- Analysis	/			
- Calculations	/			
- Data Reduction	/			
- Report Generation	/			
- Data Review	/			
- Preventive Maintenance	/			
Quality Control	/			
Corrective Action	/			
Pollution Prevention	/			
Waste Management	/			
Supplementary Notes	/			
References	/			
Appendices	/			

This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

KHOA VAN VU *Kho Van Vu*

Date:

6/16/15

## STANDARD OPERATING PROCEDURES

**SEMIVOLATILE ORGANICS BY GC/MS SIM**

SOP No.:	<u>EMAX-8270SIM</u>	Revision No. <u>2</u>	Date: <u>05-Jul-11</u>
Prepared By:	<u>Souzan Greas</u> <i>Souzan Greas</i>		Date: <u>07-1-11</u>
Approved By:	<u>Kenette Pimentel</u> <i>KP</i>		Date: <u>07.01.11</u>
	QA Manager		
Approved By:	<u>Caspar Pang</u> <i>Caspar Pang</i>		Date: <u>07-01-11</u>
	Laboratory Director		
<b>Control Number:</b>			<u>8270SIM-02-</u>

**1.0 SCOPE AND APPLICATION**

- 1.1. This method is used to determine the trace levels of semi-volatile organic compounds extracted from many types of solid waste matrices, soil and water samples. Analytes that are listed in Table 7 were determined when this SOP was established. Additional analytes may be added upon completion of similar validation as the analytes that are listed in Table 7. This SOP is an adaptation of method SW846 8270D. Since 8270D is an update and enhancement of 8270C, this SOP is also applicable to 8270C.

**2.0 SUMMARY OF METHOD**

- 2.1. Samples are extracted with methylene chloride. Extracts are concentrated and appropriate clean-up procedure is applied if necessary.
- 2.2. Internal standards are added to an aliquot of the final extract and are qualitatively and quantitatively analyzed by gas chromatography equipped with mass spectrometry (GC/MS).
- 2.3. **Interference**
- 2.3.1. Solvents, reagents, glassware, and other sample processing devices are all possible sources of artifacts and/or interferences to sample analysis. Hence, quality control of chemicals and proper decontamination of re-usable glassware and proofing the analytical instrument to be free from contamination shall be observed.
- 2.3.2. Raw GC/MS data from all blanks, samples and spikes must be evaluated for interference. Determine source of interference in the preparation and/or cleanup of the samples and perform corrective action to eliminate the problem.
- 2.3.3. Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carry-over, rinse the sample syringe with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.
- 2.3.4. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

**3.0 DETECTION LIMITS**

- 3.1. **Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)**
- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Refer to Table 7 for established limits.

## STANDARD OPERATING PROCEDURE

**SEMIVOLATILE ORGANICS BY GC/MS SIM**


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**4.0 DYNAMIC RANGE**

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.
- 4.3. The dynamic range established for this method are:

<u>Water (µg/L)</u>	<u>Soil (µg/kg)</u>
1 - 100 µg/L	5 - 3300 µg/kg

**5.0 SAMPLE HOLDING TIME AND PRESERVATION****5.1. Sample Preservation**

- 5.1.1. Store water and soil samples at  $\leq 6^{\circ}\text{C}$  away from light without freezing.
- 5.1.2. Store all extracts at  $\leq 6^{\circ}\text{C}$  without freezing.

**5.2. Holding Time**

- 5.2.1. Extract water samples within 7 days from sampling date.
- 5.2.2. Extract soil samples within 14 days from sampling date.
- 5.2.3. Analyze all extracts within 40 days from extraction completion date.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-3510 Extraction of Organic Compounds by Separatory Funnel
- 6.2. EMAX-3520 Extraction of Organic Compounds by Continuous Liquid/Liquid Extraction
- 6.3. EMAX-3540 Soxhlet Extraction
- 6.4. EMAX-3550 Extraction of Organic Compounds from Solid Samples by Pulse Sonication
- 6.5. EMAX-3580 Waste Dilution
- 6.6. EMAX-3640 Clean Up, GPC
- 6.7. EMAX-DM01 Data Flow and Review
- 6.8. EMAX-QA05 Training
- 6.9. EMAX-QA04 Detection Limit Study
- 6.10. EMAX-QA08 Corrective Action
- 6.11. EMAX-QC01 Quality Control for Chemicals
- 6.12. EMAX-QC02 Analytical Standard Preparation

## STANDARD OPERATING PROCEDURE

**SEMIVOLATILE ORGANICS BY GC/MS SIM**


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SOP No.: EMAX-8270SIM Revision No. 2 Date: 05-Jul-11

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- 6.13. EMAX-QC07 Glassware Cleaning
- 6.14. EMAX-SM01 Sample Management
- 6.15. EMAX-SM03 Waste Disposal
- 6.16. EMAX-SM04 Analytical and QC Sample Labeling

**7.0 SAFETY**

- 7.1. Read all MSDS for all chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. Place all wastes generated during analytical process in the designated satellite waste containers. Endorse these wastes to the waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS AND REAGENTS****8.1. Instruments and Supplies**

Gas Chromatography	Agilent Technologies 7890A with split/splitless injection, Shimadzu GC-17A, or equivalent
Mass Spectrometer	Agilent Technologies 5975C MSD or Shimadzu GCMS – GP 5000 capable of scanning from 1.6 to 1050 amu every 1 second using 70 volts electrode energy in the electron impact ionization mode or equivalent
GC/MS Interface	Capillary-direct into the mass spectrometer source or equivalent
Chromatographic Column	ZB-5MS (20m x 0.18 mm x 0.32 µm) or equivalent
Data System	MS-ChemStation with Enviroquant software or equivalent
GC Autosampler	Agilent Technologies 7683B series injector or Shimadzu AOC-20i capable of direct injection of 1 µl and 10 µl of extract.
Gases	Ultra high purity helium
Syringes	10 µl, 25 µl, 50 µl, 100 µl, 250 µl, 500 µl and 1000 µl syringe Hamilton 202N or equivalent
Vials	Autosampler vials with teflon lined septa

**8.2. Chemicals and Reagents**

Solvents	Methylene chloride pesticides grade, high purity methanol
Reagents	Na <sub>2</sub> SO <sub>4</sub> reagent grade

## STANDARD OPERATING PROCEDURE

**SEMIVOLATILE ORGANICS BY GC/MS SIM**

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**9.0 STANDARDS****9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX-SM04, respectively.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

**9.2. Stock Standard**

- 9.2.1. Purchase stock standards as certified solutions from AccuStandard or other reputable vendor (refer to Table 1 for the listing of all certified solutions). The standard is expected to be at 96% purity. Read vendor's note if correction has been applied to certified values otherwise corrections must be applied.
- 9.2.2. Transfer the stock standard solutions into 2 ml amber vial with Teflon lined screw caps and store at -10°C to -20°C.

**9.3. Intermediate Standard**

- 9.3.1. Using stock standard solutions, prepare the intermediate standard in Methylene Chloride according to table 1.

**9.4. Internal Standard**

- 9.4.1. The internal standard shall include 1,4-Dichlorobenzene-d<sub>4</sub>, Phenanthrene-d<sub>10</sub> and Perylene-d<sub>12</sub> in methylene chloride solution.
- 9.4.2. Purchase internal standard solutions as certified solution from AccuStandard or other reputable vendor at 4,000 µg/ml.
- 9.4.3. Prepare a 10 ml of 2,000 µg/ml of working internal standard from 4,000 µg/ml (refer to Table 2). Transfer the solution in a properly labeled 10 ml amber vial and store in -10°C to -20°C.

**9.5. GC/MS Tuning**

- 9.5.1. The tuning standard shall include decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol, and Benzidine.
- 9.5.2. Purchase tuning standard solution as certified standard at 1000 µg/ml.
- 9.5.3. Prepare a 500 µl of 50 µg/ml of working standard tuning solution (refer to Table 2). Transfer the solution in a 1 ml amber vial and store in -10°C to -20°C.

**9.6. Surrogate Standard**

- 9.6.1. Purchase surrogate stock standards as certified standard.
  - 9.6.1.1. The acid surrogate mixture includes Phenol-d<sub>5</sub>, 2-Fluorophenol and 2,4,6-Tribromophenol at 150 µg/ml.
  - 9.6.1.2. The basic neutral surrogate mixture includes Nitrobenzene-d<sub>5</sub>, 2-Fluorobiphenyl, Terphenyl-d<sub>14</sub> and 1,2-Dichlorobenzene-d<sub>4</sub> at 50 µg/ml.
- 9.6.2. For typical extraction [soil: 30 g - 2 ml or water: 1000 ml - 2 ml], add 0.4 ml of surrogate spiking standard to the sample prior to extraction. Spike volume may be adjusted to normalize with the final extract and yield the same concentration.

## STANDARD OPERATING PROCEDURE

**SEMIVOLATILE ORGANICS BY GC/MS SIM**

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SOP No.: EMAX-8270SIM Revision No. 2 Date: 05-Jul-11

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**9.7. Calibration Standard**

9.7.1. Prepare working standard solutions for initial calibration and daily calibration (refer to Table 2 for details). Transfer the solutions in 1 ml amber vial and store them at  $\leq 6^{\circ}\text{C}$  without freezing.

**9.8. ICAL Verification Standard (Second Source Verification) (ICV)**

9.8.1. Purchase a certified ICV standard from a different vendor. The ICV standard contains the same list of compounds as the stock standard (refer to Table 1b for the standard mix and the corresponding vendors).

9.8.2. Prepare a 500  $\mu\text{l}$  of 25  $\mu\text{g}/\text{ml}$  check standard solution (refer to Table 2). Transfer the solution in a properly labeled 1 ml amber vial and store at  $\leq 6^{\circ}\text{C}$  without freezing.

**9.9. LCS/MS Spiking Standards**

9.9.1. Purchase spiking standards as certified solutions. The spiking solution may be from the same source as the initial calibration standard<sup>1</sup>.

9.9.2. Spike MS/MSD/LCS/LCD samples with 0.2 ml of full spiking solution prior to sample extraction.

9.9.3. Spike volume may be adjusted to normalize with the final extract volume and yield the same concentration.

**10.0 PROCEDURES****10.1. Sample Preparation**

10.1.1. For aqueous samples, refer to EMAX-3510 or EMAX-3520. Check the extraction log for presence of residual chlorine.

10.1.2. For solid samples, refer to EMAX-3550 or EMAX-3540.

10.1.3. For waste samples, refer to EMAX-3580.

10.1.4. After extraction, examine the color and consistency of the extract. If the extract appears to be opaque and/or viscous, it is advisable to perform extract cleanup preferably GPC. Refer to EMAX-3640.

**10.2. Instrument Parameters**

10.2.1. Set the instrument parameters as suggested in Table 3. Fine tune the instrument to obtain optimum instrument condition.

10.2.2. Print and display current condition on the instrument for easy access when performing daily instrument routine check.

10.2.3. In the event that instruments parameters necessitate a change, replace the instrument parameter print-out with the new parameter setup and archive the previous instrument parameters in the instrument maintenance log.

10.2.4. Set injection volume to 1  $\mu\text{l}$  to 2  $\mu\text{l}$ .

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<sup>1</sup> SW846 Method 8270D, Section 7.9

## STANDARD OPERATING PROCEDURE

**SEMIVOLATILE ORGANICS BY GC/MS SIM**

SOP No.: EMAX-8270SIM Revision No. 2 Date: 05-Jul-11

**10.3. Calibration**

10.3.1. Set GC/MS operating condition as described in Section 10.2.

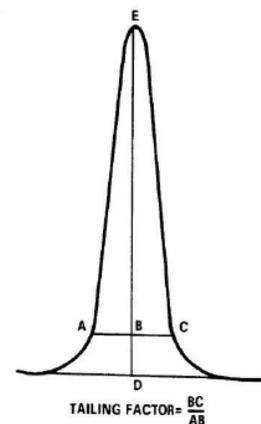
10.3.2. Perform Tune Check

10.3.2.1. Analyze a solution containing 50 µg/ml of tuning standard working solution, DFTPP, DDT, benzidine and pentachlorophenol.

10.3.2.2. Evaluate the tune check by averaging of three scans (the peak apex scan and the scan immediately preceding and the scan immediately following the apex). Apply a background subtraction using a single scan no more than 20 scans prior to the elution of DFTPP. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP. Use the DFTPP mass intensity criteria in the manufacturer's instructions as primary tuning acceptance criteria otherwise refer to Table 4 for acceptance criteria.

10.3.2.3. Evaluate column performance and injection port inertness using the data acquisition software.

- Degradation of DDT to DDE and DDD must be less than 20% based on area obtained from the total ion chromatogram.
- Benzidine and Pentachlorophenol must be present at their normal responses. Evaluate the tailing factor of benzidine and pentachlorophenol. Tailing factor should not be > 2. Refer to the attached Figure for peak evaluation.



Example calculation: Peak Height = DE = 100 mm  
 10% Peak Height = BD = 10 mm  
 Peak Width at 10% Peak Height = AC = 23 mm  
 AB = 11 mm  
 BC = 12 mm  
 Therefore: Tailing Factor =  $\frac{12}{11} = 1.1$

10.3.2.4. If tune check is non-compliant, refer to Section 12 for corrective action.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs:

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Optimize the instrument condition prior to ICAL:

- Ensure that instrument parameters are set up properly
- Ensure that there is no evidence of leak
- Ensure that instrument maintenance as scheduled is performed
- Ensure that instrument tune check and column performance is not indicative that it is at the threshold of failing the acceptance criteria

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10.3.3.3. Analyze a multi-point initial calibration curve as suggested in Figure 3 after a valid tune check.

10.3.3.4. Generate a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRF<sub>m</sub>), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq-10.5.1.1, Eq-10.5.1.2, Eq-10.5.1.5 and Eq-10.5.1.6, respectively.

10.3.3.5. **Evaluate the ICAL Acceptance**

10.3.3.5.1. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary.

10.3.3.5.2. Evaluate retention time of each analyte with respect to the nearest internal standard. The relative retention time (RRT) of each analyte should agree within  $\pm 0.06$  RRT units.

10.3.3.5.3. Check the response factors for each analyte as suggested in Table 6. Meeting the minimum response factor is indicative that the analytes are behaving as expected.

10.3.3.5.4. Check RSD and correlation coefficient. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform necessary instrument maintenance and repeat calibration. Refer to 10.3.3.2., Section 12 for corrective action.

10.3.3.6. **Application of ICAL Curve for Quantitation**

10.3.3.6.1. If RSD is  $\leq 20\%$  average response factor may be applied.

10.3.3.6.2. Apply Inverse Weighting Factor ( $1/y$  or  $1/y^2$ ;  $y$  being the instrument response) if it is determined to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.99$ .

10.3.3.6.3. Apply linear least squares regression if past experience or prior knowledge of instrument response is known to be the best fit for specific analytes. This approach may be applied to any analyte including analyte that has RSD of  $\leq 20\%$  and correlation coefficient of  $\geq 0.99$ .

10.3.3.6.4. It may be appropriate to force the regression through zero for specific analytes<sup>2</sup>. When exercising this option (as included in the data acquisition software), make sure that the origin (0,0) is not included as a calibration point but rather the intercept is set to zero. This option shall only be applied if the curve favors better accuracy of quantitation.

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<sup>2</sup> SW846 Method 8000B, Section 7.5.3

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10.3.3.6.5. Re-quantitate the calibration point near or at LOQ and calculate for percent recovery. Percent recovery within  $\pm 30\%$ <sup>3</sup> of their expected values is indicative that calibration accuracy at the lower calibration range is good.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Verify the concentration of the ICAL by analyzing the ICV from a secondary source (refer to Table 2 for standard preparation). Result should be within  $\pm 30\%$  of expected value unless otherwise specified by the project.

10.3.4.2. Evaluate the ICV for minimum response factor, internal standard retention time and internal standard response as described in 10.3.5.2.

10.3.4.3. If any of above criteria is non-compliant, refer to Section 12 for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC (refer to Table 2 for standard preparation) to check the validity of the ICAL every 12 hour shift unless otherwise specified by the project.

10.3.5.2. **DCC Evaluation**

- Check that RF is  $\geq$  minimum response factor listed in Table 6.
- Check that all analytes are  $\leq 20\%$  D (% Difference for ARF and %Drift for other quantitation technique) unless otherwise specified by the project.
- Check Internal Standard Retention time. Expected retention time is within  $\pm 30$  seconds from that of the midpoint of the ICAL.
- Check Internal Standard Response. Expected response of Extracted Ion Current Profile (EICP) is no greater than a factor of two (-50% to +100%).
- If any of the above criteria is non-compliant, refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Extract Preparation

10.4.1.1. Allow extracts to equilibrate with room temperature.

10.4.1.2. Measure 300  $\mu$ l of extract, transfer into an autosampler vial.

10.4.1.3. Add 6  $\mu$ l of 2000 ng/ $\mu$ l of internal standard (refer to 9.4.3).

10.4.1.4. Seal the vial with Teflon-lined septa cap.

10.4.2. Analytical Sequence

10.4.2.1. Analyze instrument blank.

10.4.2.2. Analyze DFTPP and evaluate tuning.

10.4.2.3. Analyze DCC and check ICAL validity.

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<sup>3</sup> SW846 Method 8270D, Section 11.4.5.6

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10.4.2.4. Analyze Method Blank.

10.4.2.5. Analyze Lab Control Sample and Lab Control Sample Duplicate (optional).

10.4.2.6. Analyze matrix spikes (MS/MSD) as per project requirement.

10.4.2.7. Analyze samples to a maximum of 12-hours from the time of DFTPP injection.

10.4.3. Sample Result Evaluation

10.4.3.1. **Check QC criteria**

- Check method blank. If result is non-compliant and analyte in question is not detected in any sample or contamination is < 10X of the sample concentration, results maybe reportable. Verify with the PM if results can be reported.
- Check surrogate recoveries against PSR. In the absence of PSR, default to EMAX QC limits.
- Check concentration of target analytes. Dilute and re-analyze samples having result(s) exceeding calibration range unless otherwise specified by the project. Maintain IS concentration on diluted samples.
- Check Internal Standard Response. Expected response of EICP is no greater than a factor of two (-50% to +100%).
- Check Internal Standard Retention Time. Expected retention time is within  $\pm$  30 seconds of the ICAL midpoint.
- If any of the above checkpoints indicate a problem, re-analysis is required.

10.4.3.2. **Qualitative Identification**

- The intensities of the characteristic ions must maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
- Check the chromatogram for possible misidentified analytes. Manually integrate the peak if necessary in accordance to EMAX-DM01.
- Investigate visible peaks in the chromatogram that were not identified in the data output. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification<sup>4</sup> (TIC).
- Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting.

10.4.3.3. **Quantitation**

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<sup>4</sup> Library search is performed only when indicated in the PSR.

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- Apply the appropriate quantitation method (refer to Section 10.5.3) to calculate the concentration of any positively identified target analyte. Apply the sample preparation and dilution factor to calculate for the final concentration of the sample.

**10.4.3.4. Manual Integration**

10.4.3.4.1. Refer to EMAX-DM01 for details of manual integration.

**10.4.3.5. Dealing with Carryover**

- Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction. Otherwise, clean up the system and re-analyze the sample suspected to contain carryover.

**10.5. Calculations****10.5.1. Initial Calibration****10.5.1.1. Calculate for Relative Response Factor (RRF)**

$$RRF = \frac{(AX)(CIS)}{(AIS)(CX)} \quad \text{Eq.-10.5.1.1}$$

where:

*AX* – Area of characteristic ion for the compound being measured

*AIS* – Area of characteristic ion for the specific internal standard

*CX* – Concentration of the compound being measured

*CIS* – Concentration of the specific internal standard

**10.5.1.2. Calculate for Average Relative Response Factor (RRF<sub>m</sub>)**

$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq.-10.5.1.2}$$

where:

*RRF<sub>m</sub>* – average response factor

$\sum RRF$  – summation of response factors

*n* – number of measurements

**10.5.1.3. Calculate for Least Square Linear Regression**

$$y = ax + b \quad \text{Eq.-10.5.1.3}$$

where:

*y* – Response Ratio (*AX/AIS*)

*x* – Amount Ratio (*CX/CIS*)

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 $a - x_1 = \text{slope of the line}$ 

$$a = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

where:

 $\bar{x}$  – Average of amount ratios $\bar{y}$  – Average of response ratios $b = x_0 = \text{intercept of the line}$ 

$$b = \bar{y} - a * \bar{x}$$

**10.5.1.4. Calculate for Inverse Weighting Factor**

$$y = ax + b$$

Eq.-10.5.1.4

where:

 $y$  – Response Ratio (AX/AIS) $x$  – Amount Ratio (CX/CIS) $a - x_1 = \text{slope of the line}$ 

$$a = \frac{\sum [(x - x_a)(y - y_a)]}{\sum (x - x_a)^2}$$

where:

$$x_a = \sum [x(1/x) / \sum (1/x)]$$

$$y_a = \sum [y(1/x) / \sum (1/x)] \text{ or}$$

$$x_a = \sum [x(1/x^2) / \sum (1/x^2)]$$

$$y_a = \sum [y(1/x^2) / \sum (1/x^2)]$$

 $b = x_0 = \text{intercept of the line}$ 

$$b = y_a - a * x_a$$

**10.5.1.5. Calculate for Standard Deviation**

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}}$$

Eq.-10.5.1.5

where:

 $SD$  – standard deviation

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$x_i$  – result at  $i^{\text{th}}$  measurement

$\bar{x}$  – mean

$n$  – number of measurements

10.5.1.6. **Calculate for % relative standard deviation (%RSD)**

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq.-10.5.1.6}$$

where:

$SD$  – standard deviation

$RRF_m$  – average response factor

10.5.2. Calibration Check/Continuing Calibration

10.5.2.1. **Calculate Percent Difference (%D) when RRF<sub>m</sub> is used for quantitation**

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq.-10.5.2.1}$$

where:

$RRF_c$  – response factor from continuing calibration standard

$RRF_m$  – average response factor

10.5.2.2. **Calculate Percent Deviation (%D<sub>t</sub>) when applied calculation is other than ARF**

$$\%D_t = \frac{|T_t - T_f|}{T_t} * 100\% \quad \text{Eq.-10.5.2.2}$$

where:

$T_t$  – true value of standard in µg/L

$T_f$  – found value of standard in µg/L

10.5.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.5.3.1. **Water Samples**

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \quad \text{Eq.-10.5.3.1}$$

where:

$A_x$  – area of characteristic ion for the compound to be measured

$I_s$  – amount of internal standard added

$V_e$  – extract final volume from sample extraction, usually 1 ml

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$$DF - \text{dilution factor} = \frac{\text{aliquot}(\mu\text{l}) + \text{solvent}(\mu\text{l})}{\text{aliquot}(\mu\text{l})}$$

$A_{is}$  – area of characteristic ion for the internal standard

$RRF_m$  – average response factor

$V_i$  – volume of extract injected in  $\mu\text{l}$ , usually 1  $\mu\text{l}$

$V_t$  – volume of water extracted in ml, usually 1000 ml

**10.5.3.2. Soil/Sediment Samples (Dry weight basis)**

$$\text{Concentration}(\mu\text{g} / \text{Kg}) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \quad \text{Eq.-10.5.3.2}$$

where:

$A_x$  – area of characteristic ion for the compound to be measured

$I_s$  – amount of internal standard injected in ng

$V_e$  – volume of extract in ml, usually 1 ml<sup>5</sup>

$$DF - \text{dilution factor} = \frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$$

$A_{is}$  – area of characteristic ion for the internal standard

$RRF_m$  – average response factor

$V_i$  – volume of extract injected in  $\mu\text{l}$ , usually 1  $\mu\text{l}$

$W_s$  – wet soil weight in kg

$$DW - \% \text{ solid} = \frac{100 - \% \text{moisture}}{100}$$

**10.5.3.3. Calculation for results subjected to cleanup by GPC**

$$\text{Concentration}(\mu\text{g} / \text{Kg}) = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} * \frac{V_{bg}}{V_{ig}} \quad \text{Eq.-10.5.3.3}$$

where:

$A_x$  – area of characteristic ion for the compound to be measured

$I_s$  – amount of internal standard injected in ng

$V_e$  – volume of extract in ml

$$DF - \text{dilution factor} = \frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$$

<sup>5</sup> For extracts subjected to GPC  $V_i = 0.5$  ml

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$A_{is}$  – area of characteristic ion for the internal standard

$RF_m$  – average response factor

$V_i$  – volume of extract injected in  $\mu\text{l}$ , usually 1  $\mu\text{l}$

$W_s$  – wet soil weight in kg

$$DW - \% \text{ solid} = \frac{100 - \% \text{ moisture}}{100}$$

$V_{bg}$  – total volume of extract before GPC clean-up in ml

$V_{ig}$  – injected volume of extract to GPC in ml

10.5.4. Base all sample result calculations on the ICAL curve, e.g., area ratio of  $A_x/A_{is}$  versus concentration using inverse weighting factor fitted to the initial calibration is also used for determination of sample concentration.

10.5.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.5.5.1. The area “ $A_x$ ” and “ $A_{is}$ ” are derived from total ion chromatogram. “ $A_{is}$ ” refers to the closest internal standard (IS) free of interference.

10.5.5.2. RRF of the TIC is 1.

10.5.6. Accuracy and Precision

10.5.6.1. **Percent Recovery**

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100 \quad \text{Eq.-10.5.6.1}$$

where:

$C_f$  – concentration found

$C$  – concentration of sample (use 0 for LCS)

$C_s$  – concentration of spike

10.5.6.2. **Relative Percent Difference (%RPD)**

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.5.6.2}$$

where:

$RPD$  – Relative Percent Difference

$C_1$  – Measured concentration of the first sample aliquot

$C_2$  – Measured concentration of the second sample aliquot

10.5.7. DDT Degradation

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$$\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}} (100) \quad \text{Eq.-10.6.7}$$

where:

*%B* – percent breakdown

*ADDD* – area of DDD

*ADDE* - area of DDE

*ADDT* - area of DDT

**10.6. Data Reduction**

- 10.6.1. Make a copy of the analytical run log and highlight the data to be reported.
- 10.6.2. Print a copy of the raw data and the QC report.
- 10.6.3. Check that all positively identified analytes are within the calibration range.
- 10.6.4. Collate the reportable raw data separating the QC results from the sample results.
- 10.6.5. Keep all other data generated with the analytical folder marked with "For Record Only".

**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WDB1C.exe
- 10.7.2. Generate the sample results using F1NV3C.exe
- 10.7.3. Generate the QC summary using QCV3CN.exe
- 10.7.4. Generate the Instrument Performance Check (ICAL and DCC for 8270C) using F5SVTEST.exe
- 10.7.5. Generate the Instrument Performance Check (ICAL and DCC for 8270D) using F5SVN.exe
- 10.7.6. Generate the IS and RT Summary using F8SV.exe
- 10.7.7. Generate Lab Chronicle using LABCHRN1.exe
- 10.7.8. Generate the Case Narrative using CN1.exe

**10.8. Data Review**

- 10.8.1. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
  - Case Narrative
  - Lab Chronicle
  - Sample Results
  - Method Blank Results
  - LCS/LCSD Summary
  - MS/MSD Summary
  - Instrument Performance Check (ICAL)
  - ICAL Summary

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- ICV Summary
- Instrument Performance Check (DCC)
- IS and RT Summary
- DCC Summary
- Analytical Run Log
- Sample Preparation Log
- Non-Conformance Report (if any)

10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- Check internal standard area. They should be within -50 to +100% of the ICAL midpoint to be acceptable, otherwise follow PSR.
- Check retention time of each IS to the ICAL midpoint. They should be within  $\pm 30$  seconds to be acceptable, otherwise follow PSR.
- Check surrogate recoveries against Project Specific Requirements (PSR). In the absence of PSR, default to in-house QC limits.
- Check concentrations of target analytes if calibration range is exceeded.
- If any of the above checkpoints indicate a problem, re-analysis is required.

10.8.3. Review the case narrative and check that it accurately describes what transpired in the analytical process. Edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.8.4. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Refer to form 8270SIMFM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back to control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
Autosampler	Inspect and clean syringe. Check autosampler response.	Daily prior to analysis
Vacuum System Verification	Verify pressure. Perform system tune check.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Check instrument performance	Daily prior to analysis

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	(e.g., Daily calibration check, instrument blank, DDT/Endrin breakdown).	
Source Cleaning	Remove and clean the Mass Spec ion source.	Every 6 months or as necessary
Vacuum System Maintenance	Inspect vacuum pumps, and replace mechanical/diffusion pump oil	Every 6 months
Documentation	Record maintenance in instrument service logs.	Daily prior to analysis to include services done by third party.

**11.0 QUALITY CONTROL****11.1. Sample Preparation Batch QC**

- 11.1.1. Analyze Method Blank (MB) to demonstrate that preparation of sample was free from contamination.
- 11.1.2. Analyze Lab Control Sample (LCS) to assess preparative batch accuracy.
- 11.1.3. Analyze Matrix Spike (MS/MSD) to assess matrix interference (when required by the project) and a maximum of 20 field samples of similar matrix.
- 11.1.4. In the absence of MS/MSD prepare LCS/LCSD to check for precision.
- 11.1.5. All lab wares used in the sample preparation shall be properly treated as specific in EMAX-QC07.
- 11.1.6. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use.
  - Verify that the spike amount is accurate by checking the record.
  - If LCS is within acceptance criteria and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the problem matrix interference in the case narrative.

**11.2. Analytical Batch QC**

- 11.2.1. Perform tune check to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to calibration and check for any contamination.
- 11.2.2. Perform Initial Calibration (ICAL) to establish a calibration curve for the quantification of the analytes of interest.
- 11.2.3. Establish Retention Time window position for each analyte every after ICAL for proper qualitative identification.
- 11.2.4. Perform Initial Calibration Verification (ICV) every after ICAL to verify accuracy of ICAL.
- 11.2.5. Perform Continuing Calibration Verification (CCV) every 12 hours to verify that the instrument response is reliable, and has not changed significantly from the current ICAL curve.
- 11.2.6. Evaluate Relative Retention Time for each analytes in every sample to be within  $\pm 0.06$  RRT

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units.

11.2.7. Verify Internal Standards (IS) for quantitative accuracy and that its Retention Time is within  $\pm$  30 seconds from retention time of the midpoint standard in the ICAL and EICP area is within - 50 to +100% of ICAL midpoint standard.

11.2.8. Evaluate Surrogate recovery to monitor instrument response on every sample.

**11.3. Method QC**

11.3.1. Establish Detection Limit (DL) to determine the smallest analyte concentration that can be demonstrated to be different from zero.

11.3.2. Establish Limit of Detection (LOD) to determine the smallest concentration of an analyte that can be qualitatively identified in a sample with 99% confidence level.

11.3.3. Establish Limit of Quantitation (LOQ) to determine the lowest concentration that produces a quantitative result within specified limits of precision and bias.

11.3.4. All analysts conducting this analysis must have an established Demonstration of Capability (DOC) as described in EMAX-QA05.

11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency, acceptance criteria and corrective action.

**12.0 CORRECTIVE ACTIONS**

12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.

**12.2. Sample Preparation QC**

12.2.1. If the instrument blank is non-compliant, consider the following suggestions to correct the problem:

12.2.1.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new autosampler syringe.

12.2.1.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.

12.2.1.3. Rule out preparation contamination by preparing a new instrument blank.

12.2.1.4. If the problem persists, inform the supervisor for further advice.

12.2.2. If Method Blank is non-compliant, consider the following suggestions to correct the problem:

12.2.2.1. Rule out instrument contamination by checking instrument blank.

12.2.2.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01.

12.2.2.3. Rule out glassware contamination used for extraction as described in EMAX-QC07.

12.2.2.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents.

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12.2.2.5. If the problem persists, inform the supervisor for further advice.

12.2.3. If LCS is non-compliant, perform the following suggestions to correct the problem:

12.2.3.1. If result is bias-high or bias low, check the LCS Standard by analyzing at the spike level.

12.2.3.2. If LCS Check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.

12.2.3.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.

12.2.3.4. If LCS is within acceptance then and the right amount of spike is added into sample then it is indicative of matrix interference. Discuss the probable matrix interference in Case Narrative.

12.2.4. If MS is non-compliant consider the following suggestions to correct the problem:

12.2.4.1. Verify that the spike amount added is accurate by checking the record and the micropipette calibration.

12.2.4.2. If LCS is within acceptance criteria then and the right amount of spike is added into the sample then it is indicative of matrix interference. Discuss the probable matrix interference in the Case Narrative.

### 12.3. Analytical Batch QC

#### 12.3.1. Tune Check

12.3.1.1. If tune check is non-complaint consider the following suggestion to correct the problem:

- Check the instrument settings and make sure that the instrument parameters are properly set up.
- Check gas flow.
- Perform autotune or visual optimization.
- If the problem persists, inform the supervisor for further actions.

12.3.1.2. If instrument performance is non-compliant, consider the following suggestions to correct the problem:

- Excessive degradation of DDT and/or poor chromatography demonstrated by too much tailing are indications of dirty injection port. Clean or replace the injection port. If problem persist, cut off the first 6-12 inches of the capillary column.

#### 12.3.2. Initial Calibration (ICAL)

12.3.2.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

- If RSD% is out of acceptance criteria, review result and identify presence of an outlier.

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- If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and re-analyze.
- If the highest ICAL point appears to be saturated, drop the highest point.
- If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

*Note: The lowest calibration point identifies the limit of quantitation (LOQ). Therefore, check that the LOQ is in conformance to the current projects where the ICAL will be used.*

12.3.2.2. If instrumentation problem is suspected, consider the following suggestions to correct the problem:

- Check the connections and make sure they are air-tight and perform maintenance as needed.
- Check the gas flow.
- Re-tune the MS.
- Prepare a fresh standard and repeat calibration.
- Clean the MS source and repeat the calibration.

12.3.2.3. If the problem persists, inform the supervisor for further action.

12.3.3. Initial Calibration Verification (ICV)

12.3.3.1. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Re-analyze ICV to rule out poor injection.
- If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error.
- If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration.
- If problem persist, inform the supervisor for further action.

12.3.4. Daily Calibration Check (DCC)

12.3.4.1. If DCC is non-compliant consider the following suggestions to correct the problem:

- If majority of the analyte response are low and no evidence of leak in the system is apparent, it is indicative of a poor injection or leak in the vial. Re-analyze DCC
- If problem persist, rule out standard degradation. Prepare a fresh standard and repeat DCC.

12.3.4.2. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:

- Change the liner.
- Clean the injection port.

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- Prepare a new standard.
- Cut or replace column.
- Rule out leaks by checking all connections.
- If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL.

12.3.5. Instrument Blank. If instrument blank is non-compliant, consider the following suggestions to correct the problem:

- Rule out instrument contamination.
- Rule out reagent contamination by testing each reagent used as described in EMAX-QC01.
- Rule vials and glassware contamination as described in EMAX-QC07.
- If the problem persists, inform the supervisor for further advice.

12.4. Execute a Non-Conformance Report (NCR) when the following circumstances occur:

12.4.1. If corrective action needs the function of the department; e.g. if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.

12.4.2. If corrective action needs the assistance of the project manager; e.g. if the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.

12.5. For other problems encountered, inform the supervisor immediately for further instructions.

**13.0 POLLUTION PREVENTION**

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.2. Prepare all standards in fume hoods.

13.3. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

13.4. Separate and properly identify all unused expired analytical standards for proper disposal.

**14.0 WASTE MANAGEMENT**

14.1. Practice the "Less is Better" strategies when preparing for analytical standards. This will minimize the production of surplus chemical wastes.

14.2. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM03.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group

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of chemicals that belong to the same chemical family, and which are analyzed together.

- 15.1.2. **Batch** – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.2.1. **Preparation Batch** – is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.2.2. **Analytical Batch** – is composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.3. **Detection Limit (DL)** – The lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not false positive.
- 15.1.4. **Limit of Detection (LOD)** – An estimate of the minimum amount of substance that an analytical process can reliably detect.
- 15.1.5. **Limit of Quantitation (LOQ)** – The minimum levels, concentrations or quantities of target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
- 15.1.6. **Material Safety Data Sheet (MSDS)** – is where the physical data, toxicology and safety precaution of a certain substance is listed.
- 15.1.7. **Calibration** – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.8. **Carry-over** – are contaminants retained in the instruments/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).
- 15.1.9. **Calibration Check Compounds (CCC)** – evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.
- 15.1.10. **Instrument Method** – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.11. **Instrument Blank** – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.12. **Method Blank** – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.13. **Lab Control Sample (LCS)** – is a target-analyte-free sample spiked with verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.14. **Lab Control Sample Duplicate (LCSD)** – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.
- 15.1.15. **Sample** – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.

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- 15.1.16. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.17. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.18. Matrix – is a component or form of a sample.
- 15.1.19. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.20. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.21. Re-analysis – is a repeated analysis from the same extract/digestate or sample, identified with the Lab Sample ID suffixed with "W".
- 15.1.22. Re-extract/digest – is a repeated sample preparation process identified with the Lab Sample ID suffixed with "R".
- 15.1.23. Response Factor – is the ratio of the peak area of the target compound in the sample or sample extract.
- 15.1.24. Surrogate – are compounds added to every blank, sample, matrix spike, matrix spike duplicate and standard used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be expected to be detected in environmental media.
- 15.1.25. System Performance Check Compounds (SPCC) – are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.
- 15.2. **Application of QC Procedures**
- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Semi Volatile analysis by GC/MS SIM unless otherwise other directive is specified by the project requirements.
- 15.3. **Department of Defense (DoD) Projects**
- 15.3.1. Samples from DoD sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.
- 15.4. **Department of Energy (DoE) Projects**
- 15.4.1. Samples from DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client's quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, shall be applied.

**16.0 REFERENCES**

- 16.1. "Test Methods for Evaluation of Solid Wastes", EPA SW846, Methods 8000B and 8270D.
- 16.2. EMAX Quality Systems Manual (EMAX-QS00), as updated.

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- 17.3.1. Appendix 1 Summary of Quality Control Procedures
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**17.4. Forms**

- 17.4.1. 8270SIMFS Sample Preparation Log
- 17.4.2. 8270SIMFA Analytical Run Log
- 17.4.3. 8270SIMFM Instrument Maintenance Log

**Table 1: INTERMEDIATE STANDARD PREPARATION**

**A. Primary Source: Restek, CPI or equivalent**

Compound Name	Stock / Internal Soln. Conc. (µg/ml)	Source	Preparation			Final Conc. (µg/ml)
			Aliquot (µl)	Dil. Solution	Final Vol. (ml)	
8270 Mega Mix	1000	Restek	1000	MeCl2	5	200
8270 Custom Std.	2000	CPI	500	MeCl2	5	200
Benzidine Mix	2000	Restek	500	MeCl2	5	200
Benzoic Mix	2000	Restek	500	MeCl2	5	200
Acid Surrogate Mix	7500	Restek	133.3	MeCl2	5	200
Base/Neutral Surrogate Mix	5000	Restek	200	MeCl2	5	200

**B. Secondary Source: CPI, AccuStandard or equivalent**

Compound Name	Stock Soln. Conc. (µg/ml)	Source
Custom Semi Volatile Standard	2000	CPI
8270 LCS Solution	200	CPI
8270 Internal Standard	2000	AccuStandard

**Table 2a: WORKING STANDARD CALIBRATION**

Standard Name	Intermediate Standard μl of 200 μg/ml	Internal Standard μl of 2000 μg/ml	Dil. Solvent	Final Volume (μl)	Final Conc.
1	0.19	10	MeCl <sub>2</sub>	500	0.075 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
2	0.375	10	MeCl <sub>2</sub>	500	0.15 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
3	1.25	10	MeCl <sub>2</sub>	500	0.5 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
4	2.5	10	MeCl <sub>2</sub>	500	1 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
5	5	10	MeCl <sub>2</sub>	500	2 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
6	12.5	10	MeCl <sub>2</sub>	500	5 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
7	25	10	MeCl <sub>2</sub>	500	10 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
8	50	10	MeCl <sub>2</sub>	500	20 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
9	62.5	10	MeCl <sub>2</sub>	500	25 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
10	75	10	MeCl <sub>2</sub>	500	30 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
11	100	10	MeCl <sub>2</sub>	500	40 μg/ml of Cal. Std. 40 μg/ml of Internal Std.
12	125	10	MeCl <sub>2</sub>	500	50 μg/ml of Cal. Std. 40 μg/ml of Internal Std.

**Table 2b: WORKING SECONDARY SOURCE STANDARD**

Standard Name	Soln. Conc. (μg/ml)	Preparation			Final Conc. (μg/ml)
		Aliquot (μl)	Dil. Solvent	Final Vol. (μl)	
Custom Semi Volatile Standard	2000	6.25	MeCl <sub>2</sub>	500	25
8270 LCS Solution	2000	62.5	MeCl <sub>2</sub>	500	25
8270 Internal Standard	2000	10	MeCl <sub>2</sub>	500	40

**Table 2c: WORKING INTERNAL STANDARD PREPARATION**

Standard Name	Soln. Conc. ( $\mu\text{g/ml}$ )	Preparation			Final Conc. ( $\mu\text{g/ml}$ )
		Aliquot (ml)	Dil. Solvent	Final Vol. (ml)	
Internal Standard Mix	4000	5	$\text{MeCl}_2$	10	2000

**Table 2d: WORKING GC/MS TUNING (DFTPP) STANDARD**

Standard Name	Soln. Conc. ( $\mu\text{g/ml}$ )	Preparation			Final Conc. ( $\mu\text{g/ml}$ )
		Aliquot ( $\mu\text{l}$ )	Dil. Solvent	Final Vol. ( $\mu\text{l}$ )	
GC/MS Tuning Mix	1000	25	$\text{MeCl}_2$	500	50

**Table 3: INSTRUMENT PARAMETERS**

	<b>Inst. E4 / Inst. E7</b>	<b>Inst. 052</b>
Carrier Gas	Helium at 90 psi at outlet	Helium at 90 psi at outlet
Column head pressure	15 - 35 psi at 40°C	15-35 psi at 50°C
Injection port temperature	280-300°C	280-300°C
Interface	Direct column interference at 280-300°C	Direct column interference at 280-300°C
Valve time	Split 0.2 minute	Split 0.2 minutes
<b>Oven Temperature Program</b>		
Initial Temperature	50°C/min; hold for 0min.	50°C/min; hold for 0.2min.
Rate	30°C/min to 100°C; hold for 0.0 min.; 20°C/min to 200°C; hold for 0.0 min.; 25°C/min to 320°C; hold for 2.53 min.	10°C/min to 100°C; hold for 0 min. 38°C/min to 280°C; hold for 0 min.
Run Time	14 minutes	9.94 minutes
<b>Scan Parameters</b>		
Scan start time	After solvent peak	After solvent peak
Mass range	10 to 1050 AMU	40 to 500 AMU
Multiplier voltage	1000-3000	0.7-3
Number of sampling rate	1	0.4
Threshold	300	500-1500
Tuning File	DFTPP	DFTPP

**Table 4: DFTPP KEY IONS AND ION ABUNDANCE CRITERIA**

<b>Mass</b>	<b>Ion Abundance Criteria</b>
51	10 – 80% of mass 198
68	< 2.0% of mass 69
69	present
70	< 2.0% of mass 69
127	10 – 80% of mass 198
197	< 2% of mass 198
198	Base peak, 100% relative abundance (See Note), or > 50% of mass 442
199	5 to 9% of mass 198
275	10 to 60% of mass 198
365	> 1% of mass 198
441	Present but < 24% of mass 442
442	Base peak or > 50% of mass 198
443	15 to 24% of mass 442

Note: All ion abundance MUST be normalized to m/z 198, the nominal base peak.

**Table 5: ANALYTE LISTS, QUANTITATION IONS, INTERNAL STANDARDS & SURROGATES**

Analyte	Type	Quantitation Ions	
		Primary	Secondary
1,4-Dichlorobenzene-d <sub>4</sub>	IS	152	115
N-nitrosodimethylamine	T	74.1	42.1
Phenol-d <sub>5</sub>	S	99.1	71.1
2-Fluorophenol	S	112	64
Phenol	T	94	65.1
Bis(2-Chloroethyl)ether	T	93	63
2-Chlorophenol	T	128	64
N-Nitroso-di-n-propylamine	T	70.1	42.1
Nitrobenzene-d <sub>5</sub>	S	82.1	128
2,4-Dimethylphenol	T	107	122.1
2,4-Dichlorophenol	T	162	163.9
Naphthalene	T	128.1	129.1
4-Chloro-3-methylphenol	T	107	77
2-Methylnaphthalene	T	142.1	141.1
1-Methylnaphthalene	T	142.1	141.1
2,4,6-Trichlorophenol	T	195.9	197.9
2,4,5-Trichlorophenol	T	195.9	197.9
2-Fluorobiphenyl	S	172.1	171.1
Biphenyl	T	154	153
2,6-Dimethylnaphthalene	T	156	141
Dimethylphthalate	T	163	164
Acenaphthene	T	153.1	152.1
Acenaphthylene	T	152.1	151.1
2,3,5-Trimethylnaphthalene	T	170	155
Diethylphthalate	T	149	177
Fluorene	T	166.1	165.1
Phenanthrene-d <sub>10</sub>	IS	188	93.9
Azobenzene	T	77	105
2,4,6-Tribromophenol	S	329.8	331.8
Hexachlorobenzene	T	283.8	141.9
Pentachlorophenol	T	265.8	164.9
Phenanthrene	T	178.1	152.1
Anthracene	T	178.1	152.1
Carbazole	T	167.1	139
Di-n-butylphthalate	T	149	150
1-Methylphenanthrene	T	192	165
Fluoranthene	T	202	101
Pyrene	T	202	101
Terphenyl-d <sub>14</sub>	S	244.2	122.1
Butylbenzylphthalate	T	149	91
Benzo(a)anthracene	T	228.1	113
Chrysene	T	228.1	226.1

**Table 5 (cont.): ANALYTE LISTS, QUANTITATION IONS, INTERNAL STANDARDS & SURROGATES**

Analyte	Type	Quantitation Ions	
		Primary	Secondary
bis(2-Ethylhexyl)phthalate	T	149	167
Perylene-d <sub>12</sub>	IS	264.1	130
Di-n-octylphthalate	T	149	150
Benzo(b)fluoranthene	T	149	125
Benzo(k)fluoranthene	T	252.1	125
Benzo(e)pyrene	T	252	125
Benzo(a)pyrene	T	252	125
Perylene	T	252	125
Indeno(1,2,3-cd)pyrene	T	276.1	138
Dibenzo(a,h)anthracene	T	278.1	139
Benzo(g,h,i)perylene	T	276.1	138

Notes: T – Target Compound

S – Surrogate

IS – Internal Standard

**Table 6: RECOMMENDED MINIMUM RESPONSE FACTOR**

ANALYTE	Minimum Response Factor(RF)	ANALYTE	Minimum Response Factor(RF)
N-nitrosodimethylamine	0.010	Hexachlorobenzene	0.100
Phenol	0.800	Pentachlorophenol	0.050
Bis(2-chloroethyl)ether	0.700	Phenanthrene	0.700
2-Chlorophenol	0.800	Anthracene	0.700
N-Nitroso-di-n-propylamine	0.500	Carbazole	0.010
2,4-Dimethylphenol	0.200	Di-n-butyl phthalate	0.010
2,4-Dichlorophenol	0.200	1-Methylphenanthrene	0.010
Naphthalene	0.700	Fluoranthene	0.600
4-Chloro-3-methylphenol	0.200	Pyrene	0.600
2-Methylnaphthalene	0.400	Butyl benzyl phthalate	0.010
1-Methylnaphthalene	0.010	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis-(2-ethylhexyl)phthalate	0.010
Biphenyl	0.010	Di-n-octyl phthalate	0.010
2,6-Dimethylnaphthalene	0.010	Benzo(b)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(k)fluoranthene	0.700
Acenaphthylene	0.900	Benzo(e)pyrene	0.010
Acenaphthene	0.900	Benzo(a)pyrene	0.700
2,3,5-Trimethylnaphthalene	0.010	Perylene	0.010
Diethyl phthalate	0.010	Indeno(1,2,3-cd)pyrene	0.500
Fluorene	0.900	Dibenz(a,h)anthracene	0.400
Azobenzene	0.010	Benzo(g,h,i)perylene	0.500

Notes:

1. PSR supersedes Table 6.
2. Other analytes not listed above must have a minimum response factor of 0.01.
3. Table 6 is from SW846 Method 8270D, Table 4.

**Table 7: ESTABLISHED DL, LOD AND LOQ**

Parameter	Water (µg/l)			Soil (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
Acenaphthylene	0.05	0.1	0.2	1.25	2.5	5
Anthracene	0.05	0.1	0.2	1.25	2.5	5
Azobenzene	0.05	0.1	1	1.25	2.5	5
Benzo(a)anthracene	0.09	0.1	0.2	2.45	2.5	5
benzo(a)pyrene	0.05	0.1	0.2	1.25	2.5	5
Benzo(b)fluoranthene	0.05	0.1	0.2	1.25	2.5	5
Benzo(e)pyrene	0.05	0.1	0.2	1.25	2.5	5
Benzo(g,h,i)perylene	0.05	0.1	0.2	1.25	2.5	5
Benzo(k)fluoranthene	0.05	0.1	0.2	1.25	2.5	5
Biphenyl	0.05	0.1	1	1.25	2.5	5
bis(2-chloroethyl)ether	0.05	0.1	1	1.5	2.5	5
bis(2-Ethylhexyl)phthalate	0.57	1	2	25	50	100
Carbazole	0.05	0.1	1	1.25	2.5	5
4-Chloro-3-methylphenol	0.05	0.1	1	2.5	5	10
2-Chlorophenol	0.05	0.1	1	2.5	5	10
Chrysene	0.06	0.1	0.2	2.2	2.5	5
Dibenzo(a,h)anthracene	0.05	0.1	0.2	1.25	2.5	5
2,4-Dichlorophenol	0.05	0.1	1	2.5	5	10
2,6-Dimethylnaphthalene	0.05	0.1	0.2	1.25	2.5	5
2,4-Dimethylphenol	0.07	0.1	1	2.5	5	10
Fluoranthene	0.05	0.1	1	1.25	2.5	5
Fluorene	0.05	0.1	0.2	1.25	2.5	5
Hexachlorobenzene	0.05	0.1	1	1.25	2.5	5
Indeno(1,2,3-cd)pyrene	0.05	0.1	0.2	1.25	2.5	5
1-Methylnaphthalene	0.05	0.1	0.2	1.25	2.5	5
2-Methylnaphthalene	0.05	0.1	0.2	1.25	2.5	5
1-Methylphenanthrene	0.05	0.1	0.2	1.25	2.5	5
Naphthalene	0.05	0.1	0.2	1.25	2.5	5
n-Nitrosodimethylamine	0.05	0.1	1	1.25	2.5	5
n-Nitroso-di-n-propylamine	0.05	0.1	1	1.25	2.5	5
Pentachlorophenol	0.4	0.5	1	18	25	50
Perylene	0.05	0.1	0.2	1.25	2.5	5
Phenanthrene	0.05	0.1	0.2	1.25	2.5	5
Phenol	0.05	0.1	1	2.5	5	10
Pyrene	0.05	0.1	0.2	1.25	2.5	5
2,4,5-Trichlorophenol	0.05	0.1	1	2.5	5	10
2,4,6-Trichlorophenol	0.05	0.1	1	2.5	5	10

**Table 7 (cont.): ESTABLISHED DL, LOD AND LOQ**

Parameter	Water (µg/l)			Soil (µg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
2,3,5-Trimethylnaphthalene	0.05	0.1	0.2	1.25	2.5	5
Phenol-d5	0.15	0.3	0.6	3.75	7.5	15
2-Fluorobiphenyl	0.05	0.1	0.2	1.25	2.5	5
Nitrobenzene-d5	0.05	0.1	0.2	1.25	2.5	5
2,4,6-Tribromophenol	0.15	0.3	0.6	3.75	7.5	15
Terphenyl-d14	0.05	0.1	0.2	1.5	2.5	5
2-Fluorophenol	0.15	0.3	0.6	3.75	7.5	15
Dimethylphthalate	0.1	0.2	0.4	2.5	5	10
Diethylphthalate	0.1	0.2	0.4	2.5	5	10
Acenaphthene	0.05	0.1	0.2	1.25	2.5	5
Di-n-butylphthalate	0.1	0.2	0.4	2.5	5	10
Butylbenzene phthalate	0.1	0.2	0.4	2.5	5	10
Di-n-octylphthalate	0.1	0.2	0.4	2.5	5	10

Note:

1. PSR supersedes Table 7
2. LOD and LOQ were established at the time this SOP is written

**Figure 1:** **PEAK EVALUATION TECHNIQUE**

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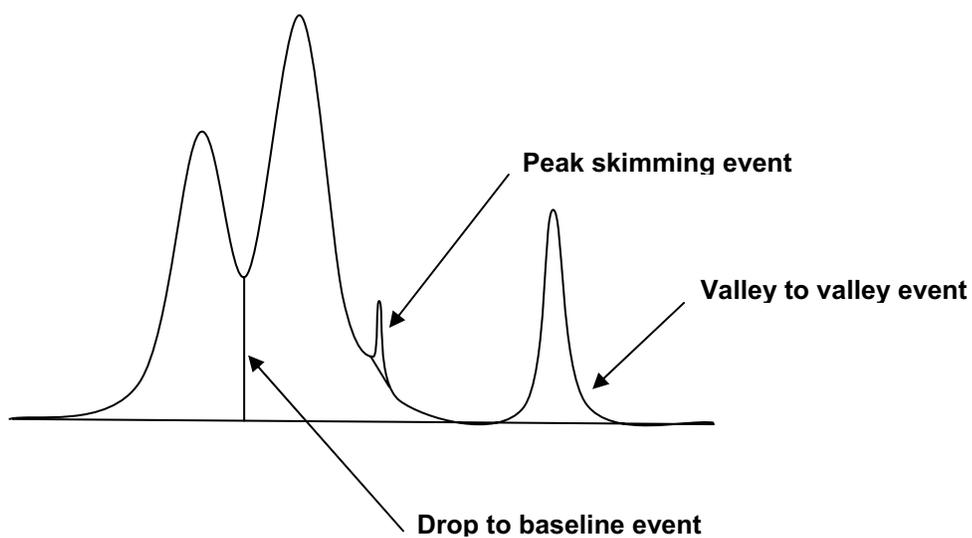
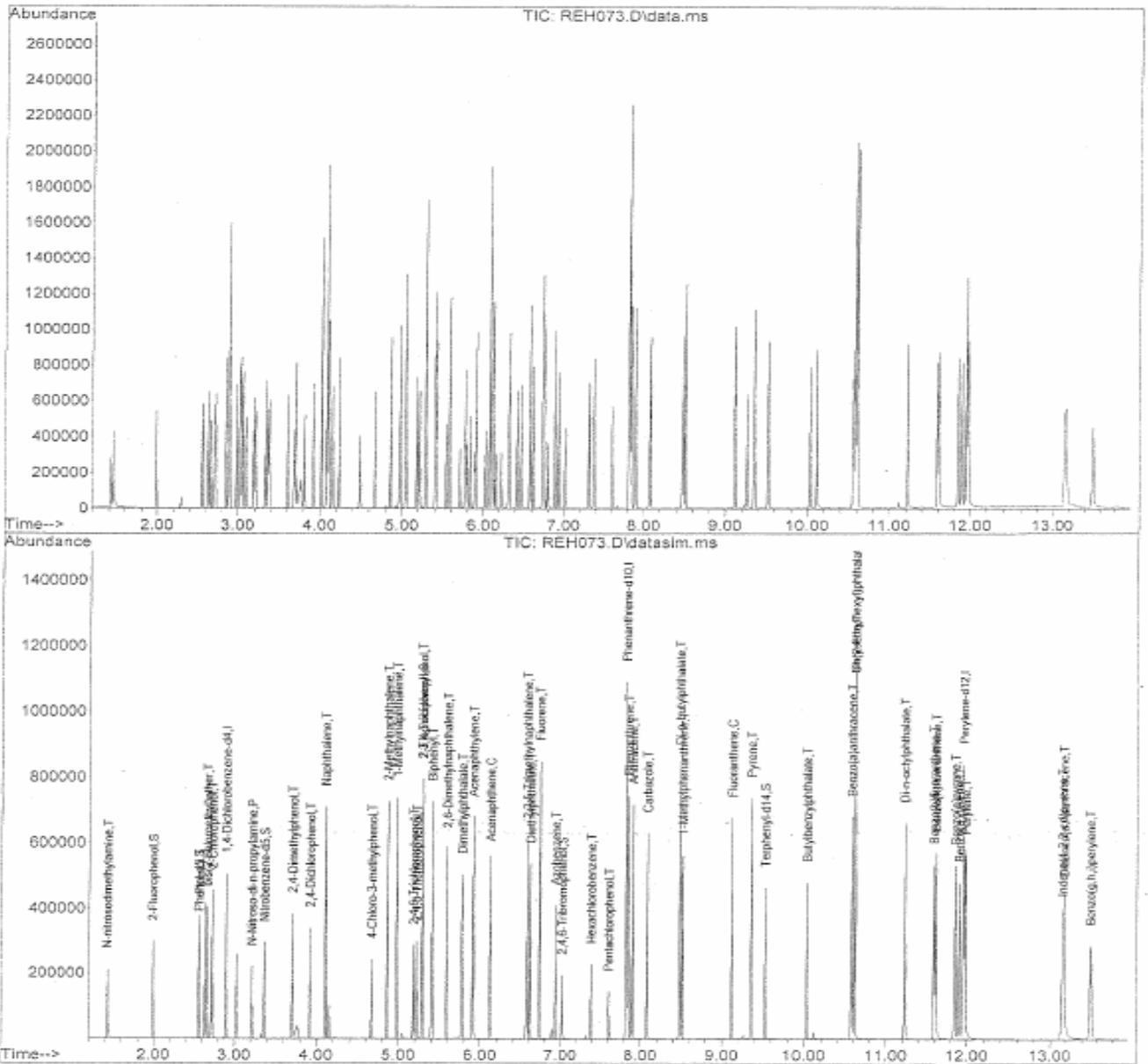


Figure 2: TYPICAL CHROMATOGRAM

Quantitation Report (QT Reviewed)  
Data File : D:\DATA\11E11\REH073.D Vial: 7  
Acq On : 11 May 2011 15:47 Operator: KV  
Sample : SVE7E11S 20PPM Inst : E7  
Misc : Multiplr: 1.00  
Integrator: RTE  
Quant Time: May 12 10:00:07 2011  
Quant Results File: SVE7E11S.RES  
Quant Method : C:\msdchem\1\METHODS\SVE7E11S.M  
Quant Title : SEMIVOLATILES - SIM  
QLast Update : Thu May 12 09:51:52 2011  
Response via : Initial Calibration  
DataAcq Meth:SVE7E11S.M



SVE7E11S.M Thu May 12 10:00:20 2011

Page: 3

Figure 3: TYPICAL ICAL SUMMARY

Instrument ID :E7  
Beginning Date/Time :05/11/11 14:33  
Spike Units :PPM  
IC File :REH072

INITIAL\_CALIBRATION - RELATIVE\_RESPONSE\_FACTOR

Column Spec :ZB-5MS ID :0.18MM  
Ending Date/Time :05/11/11 17:57  
HPChem Method :SVE7E11S

IDX	Parameters	17:075 REH080	17:330 REH079	17:205 REH078	17:02 REH077	16:44 REH076	16:22 REH075	16:10 REH074	15:20 REH073	15:05 REH072	15:30 REH071	14:40 REH070	14:30 REH069	AV RRF	% RSD	AV Rt M
1	1,4-Dichlorobenzene-d4	1	1	1	1	1	1	1	1	1	1	1	1	1	0	2.8919
2	N-nitrosodimethylamine	0.467	0.411	0.448	0.471	0.467	0.505	0.480	0.488	0.474	0.485	0.481	0.488	0.464	8.007	1.16270
3	2-Fluorophenol	1.111	1.173	1.084	1.073	1.101	1.091	1.060	1.048	1.044	1.035	1.031	1.030	1.030	0.078	1.16270
4	Phenol-d5	1.111	1.350	1.111	1.318	1.111	1.159	1.111	1.111	1.111	1.185	1.111	1.111	1.181	1.111	1.16270
5	bis(2-chloroethyl)ether	1.111	1.409	1.111	1.115	1.111	1.237	1.111	1.111	1.111	1.222	1.111	1.111	1.222	1.111	1.16270
6	N-Chlorophenol	1.111	1.312	1.111	1.025	1.111	0.970	1.111	1.111	1.111	0.933	1.111	1.111	0.933	1.111	1.16270
7	N-Nitroso-di-n-propylamine	0.001	0.423	0.001	0.385	0.001	0.587	0.001	0.001	0.001	0.293	0.001	0.001	0.341	0.001	1.16270
8	Nitrobenzene-d5	1.111	1.108	1.047	1.037	1.111	1.003	1.111	1.078	1.111	0.617	1.111	1.111	0.617	1.111	1.16270
9	2,4-Dimethylphenol	1.111	1.125	1.112	1.118	1.111	1.118	1.111	1.182	1.111	1.162	1.111	1.111	1.157	1.111	1.16270
10	2,4-Dichlorophenol	1.111	1.237	1.111	1.104	1.111	0.990	1.111	1.197	1.111	1.180	1.111	1.111	1.166	1.111	1.16270
11	Naphthalene	1.002	0.477	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
12	4-Chloro-3-methylphenol	2.896	0.890	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
13	1-Methylnaphthalene	3.000	0.736	0.610	0.590	0.485	0.485	0.485	0.485	0.485	0.485	0.485	0.485	0.485	0.485	1.16270
14	4,6-Trichlorophenol	0.000	0.783	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
15	2,2-Trichlorophenol	0.000	0.853	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
16	Fluorobiphenyl	0.660	0.320	0.147	0.147	0.147	0.147	0.147	0.147	0.147	0.147	0.147	0.147	0.147	0.147	1.16270
17	Biphenyl	1.099	0.688	0.570	0.550	0.360	0.360	0.360	0.360	0.360	0.360	0.360	0.360	0.360	0.360	1.16270
18	6-Dimethylnaphthalene	0.683	0.422	0.350	0.341	0.341	0.341	0.341	0.341	0.341	0.341	0.341	0.341	0.341	0.341	1.16270
19	Dimethylphthalate	0.355	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	0.222	1.16270
20	Acenaphthylene	0.355	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	0.776	1.16270
21	Acenaphthene	0.321	0.812	0.744	0.744	0.584	0.584	0.584	0.584	0.584	0.584	0.584	0.584	0.584	0.584	1.16270
22	1,2,3,5-Trimethylnaphthalene	0.356	0.089	0.066	0.066	0.036	0.036	0.036	0.036	0.036	0.036	0.036	0.036	0.036	0.036	1.16270
23	Diethylphthalate	0.247	0.783	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
24	Fluorene	0.211	0.853	0.874	0.867	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	0.880	1.16270
25	Phenanthrene-d10	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.16270
26	Azobenzene	0.563	0.516	0.544	0.562	0.576	0.633	0.616	0.604	0.599	0.601	0.599	0.601	0.584	0.566	0.61774
27	2,4,6-Tribromophenol	0.431	0.167	0.139	0.139	0.158	0.158	0.158	0.158	0.158	0.158	0.158	0.158	0.158	0.158	1.16270
28	Hexachlorobenzene	0.431	0.380	0.359	0.359	0.347	0.347	0.347	0.347	0.347	0.347	0.347	0.347	0.347	0.347	1.16270
29	Pentachlorophenol	0.431	0.143	0.130	0.130	0.140	0.140	0.140	0.140	0.140	0.140	0.140	0.140	0.140	0.140	1.16270
30	Phenanthrene	1.517	1.372	1.317	1.243	1.200	1.229	1.210	1.191	1.176	1.167	1.167	1.167	1.167	1.167	1.16270
31	Anthracene	1.351	1.241	1.190	1.168	1.153	1.122	1.111	1.111	1.111	1.111	1.111	1.111	1.111	1.111	1.16270
32	Carbazole	1.080	1.042	0.988	0.986	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	1.16270
33	Di-n-butylphthalate	0.073	1.019	1.021	1.017	1.079	1.079	1.079	1.079	1.079	1.079	1.079	1.079	1.079	1.079	1.16270
34	1-Methylphenanthrene	0.853	0.840	0.823	0.809	0.817	0.817	0.817	0.817	0.817	0.817	0.817	0.817	0.817	0.817	1.16270
35	Fluoranthene	1.260	1.195	1.147	1.110	1.113	1.200	1.175	1.170	1.166	1.166	1.166	1.166	1.166	1.166	1.16270
36	Pyrene	1.361	1.298	1.270	1.237	1.245	1.332	1.298	1.266	1.266	1.266	1.266	1.266	1.266	1.266	1.16270
37	Terphenyl-d14	0.362	0.798	0.810	0.793	0.833	0.833	0.833	0.833	0.833	0.833	0.833	0.833	0.833	0.833	1.16270
38	Butylbenzylphthalate	0.366	0.331	0.331	0.373	0.373	0.453	0.453	0.453	0.453	0.453	0.453	0.453	0.453	0.453	1.16270
39	Benzo(a)anthracene	1.502	1.892	1.380	1.129	1.129	1.188	1.148	1.119	1.116	1.116	1.116	1.116	1.116	1.116	1.16270
40	Chrysene	1.457	1.420	1.222	1.174	1.138	1.163	1.105	1.071	1.058	1.050	1.044	1.030	1.030	1.030	1.16270
41	bis(2-Ethylhexyl)phthalate	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.16270
42	Perylene-d12	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.16270
43	Di-n-octylphthalate	0.629	0.558	0.677	0.630	0.755	0.994	1.099	1.214	1.226	1.226	1.253	1.266	0.960	29.9	1.16270
44	Benzo(b)fluoranthene	1.126	1.039	1.094	0.988	1.003	1.112	1.091	1.130	1.119	1.082	1.089	1.089	1.081	1.081	1.16270
45	Benzo(k)fluoranthene	1.180	1.129	1.125	1.055	1.061	1.144	1.135	1.103	1.100	1.111	1.111	1.111	1.114	1.114	1.16270
46	Benzo(e)pyrene	1.285	1.161	1.140	1.035	1.035	1.111	1.108	1.098	1.099	1.087	1.087	1.087	1.087	1.087	1.16270
47	Benzo(a)pyrene	1.031	0.982	1.000	0.926	0.926	1.059	1.059	1.059	1.059	1.059	1.059	1.059	1.059	1.059	1.16270
48	Benzo(a)pyrene	1.031	1.396	1.306	1.236	1.159	1.066	1.066	1.066	1.066	1.066	1.066	1.066	1.066	1.066	1.16270
49	Indeno(1,2,3-cd)pyrene	1.207	1.242	1.117	1.076	1.087	1.221	1.233	1.223	1.234	1.234	1.234	1.234	1.234	1.234	1.16270
50	Dibenzo(a,h)anthracene	0.959	1.024	0.936	0.885	0.904	1.009	1.009	1.002	1.000	1.017	1.018	1.018	0.983	0.983	1.16270
51	Dibenzo(g,h,i)perylene	1.068	1.069	0.956	0.874	0.891	0.978	0.978	0.978	0.978	0.978	0.978	0.978	0.978	0.978	1.16270

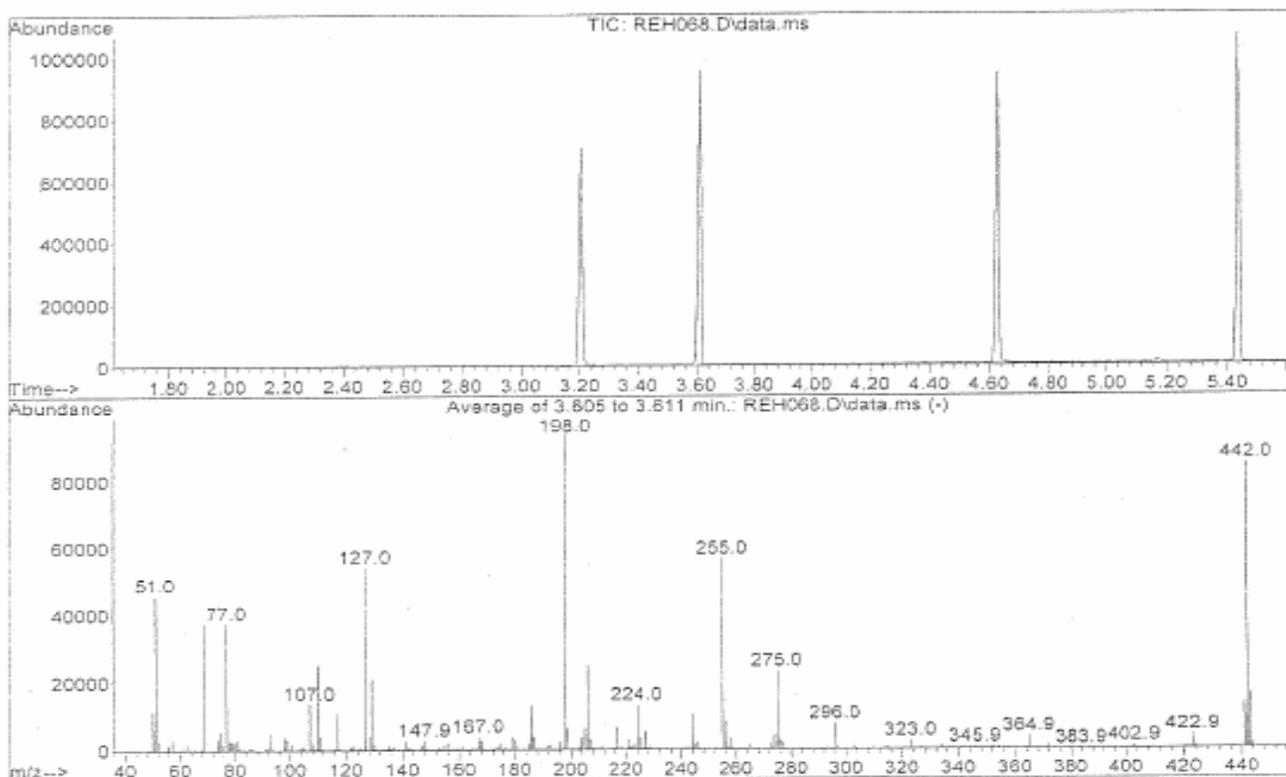
Ave\_%RSD : 7.8      Max\_%RSD : 32.3

Use Least Square Linear Regression with weighting factor of inverse concentration  
Resp\_Ratio = x0 + x1 \* Amt\_Ratio

IDX	Parameter	x0	x1	CCF
26	Diethylphthalate	-0.00358	2.99059	0.99926
41	Butylbenzylphthalate	-0.00105	1.52641	0.99888
42	Benzo(a)anthracene	-0.00277	1.11218	0.99939
44	bis(2-Ethylhexyl)phthalate	0.0470	1.7625	0.99939
46	Di-n-octylphthalate	-0.00352	1.22607	0.9978

**Figure 4: TYPICAL DFTPP TUNE SUMMARY**

Data Path : D:\DATA\11E11\  
 Data File : REH068.D  
 Acq On : 11 May 2011 14:23  
 Operator : KV  
 Sample : DFTE7E1101  
 Misc :  
 ALS Vial : 2 Sample Multiplier: 1  
 Integration File: rteint.p  
 Method : C:\msdchem\1\METHODS\DFTPPD.M  
 Title : Semivolatiles DFTPP  
 Last Update : Wed May 11 16:53:22 2011



AutoFind: Scans 717, 718, 719; Background Corrected with Scan 710

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	48.5	45907	PASS
68	69	0.00	2	1.5	559	PASS
70	69	0.00	2	0.4	152	PASS
127	198	10	80	57.4	54259	PASS
197	198	0.00	2	0.4	418	PASS
198	198	100	100	100.0	94557	PASS
199	198	5	9	6.6	6247	PASS
275	198	10	60	24.5	23205	PASS
365	198	1	100	3.9	3675	PASS
441	442	0.01	24	15.8	13420	PASS
442	198	50	100	89.6	84768	PASS
443	442	15	24	19.4	16412	PASS



Figure 5: TYPICAL CONTINUING CALIBRATION SUMMARY

CONTINUE\_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :E7  
IC\_Beginning DateTime :05/11/11 14:33  
Spike Amount :25 PPM  
CC/CV File :RFH090  
IC File :REH072  
Column Spec :ZB-5MS ID :0.18MM  
IC\_Ending DateTime :05/11/11 17:57  
HPChem Method :SVE7E11S  
Date\_Time :06/09/11 11:50

M_IDX	Parameters	CC_Con	CCX_D	CC_Resp	CCRRF	AvRRF	CC_Rtm	AvRtm	%_RSD	Co_X0	Co_X1	Co_X2	Co_Cor
1	1,4-Dichlorobenzene-d4	40.000	0	261180	1	1	2.893	2.892	0				
2	N-nitrosodimethylamine	25.609	2.4	77549	0.475	0.464	1.423	1.427	8.07				
3	2-Fluorophenol	23.348	-6.6	164332	1.007	1.078	1.992	1.989	6.33				
4	Phenol-d5	23.975	-4.1	193161	1.183	1.234	2.557	2.552	5.55				
5	Phenol	23.261	-7.0	196861	1.206	1.296	2.570	2.563	7.63				
6	bis(2-chloroethyl)ether	22.190	-11.2	148089	0.907	1.022	2.661	2.658	10.78				
7	2-Chlorophenol	24.053	-3.8	210596	1.290	1.341	2.723	2.720	5.43				
8	N-Nitroso-di-n-propylamine	25.628	2.5	102306	0.627	0.611	3.218	3.214	3.05				
9	Nitrobenzene-d5	25.568	2.3	179083	1.097	1.073	3.370	3.365	4.00				
10	2,4-Dimethylphenol	25.328	1.3	191402	1.173	1.157	3.713	3.708	3.26				
11	2,4-Dichlorophenol	25.463	1.9	193919	1.188	1.166	3.930	3.922	4.13				
12	Naphthalene	22.422	-10.3	583729	3.576	3.987	4.110	4.108	9.26				
13	4-Chloro-3-methylphenol	26.415	5.7	164622	1.008	0.954	4.681	4.673	6.30				
14	2-Methylnaphthalene	24.533	-1.9	397987	2.438	2.485	4.868	4.865	6.27				
15	1-Methylnaphthalene	22.821	-8.7	381284	2.336	2.559	4.979	4.977	6.66				
16	2,4,6-Trichlorophenol	27.175	8.7	152439	0.934	0.859	5.189	5.180	6.24				
17	2,4,5-Trichlorophenol	27.402	9.6	164294	1.006	0.918	5.226	5.216	6.35				
18	2,3,4-Trichlorophenol	27.642	10.6	144544	0.885	0.801	5.298	5.291	6.24				
19	2-Fluorobiphenyl	24.513	-1.9	488532	2.993	3.052	5.290	5.288	7.97				
20	Biphenyl	23.505	-6.0	527356	3.231	3.436	5.406	5.405	7.71				
21	2,6-Dimethylnaphthalene	24.243	-3.0	375819	2.302	2.374	5.593	5.588	5.16				
22	Dimethylphthalate	24.245	-3.0	470765	2.884	2.974	5.777	5.770	4.12				
23	Acenaphthylene	24.357	-2.6	636997	3.902	4.005	5.916	5.911	3.77				
24	Acenaphthene	23.245	-7.0	401510	2.460	2.645	6.125	6.120	8.20				
25	2,3,5-Trimethylnaphthalene	25.829	3.3	352975	2.162	2.093	6.586	6.581	4.72				
26	Diethylphthalate	25.051	0.2	490102	3.002	3.276	6.626	6.622	17.50	0.0036	2.9906		0.9996
27	Fluorene	24.472	-2.1	459608	2.816	2.876	6.741	6.737	4.52				
28	Phenanthrene-d10	40.000	0	970741	1	1	7.821	7.818	0				
29	Azobenzene	24.518	-1.9	347489	0.573	0.584	6.939	6.934	5.66				
30	2,4,6-Tribromophenol	25.284	1.1	108168	0.178	0.176	7.023	7.018	8.38				
31	Hexachlorobenzene	22.596	-9.6	193649	0.319	0.353	7.385	7.379	8.65				
32	Pentachlorophenol	27.589	10.4	114552	0.189	0.171	7.609	7.602	12.76				
33	Phenanthrene	21.607	-13.6	655171	1.080	1.249	7.850	7.843	8.46				
34	Anthracene	22.765	-8.9	664058	1.095	1.202	7.904	7.899	4.61				
35	Carbazole	23.290	-6.8	586187	0.966	1.037	8.086	8.079	2.99				
36	Di-n-butylphthalate	25.859	3.4	752057	1.240	1.198	8.486	8.481	11.71				
37	1-Methylphenanthrene	23.652	-5.4	476455	0.785	0.830	8.519	8.514	3.19				
38	Fluoranthene	24.549	-1.8	695869	1.147	1.168	9.125	9.120	3.41				
39	Pyrene	23.078	-7.7	715520	1.179	1.278	9.361	9.354	3.04				
40	Terphenyl-d14	24.082	-3.7	457518	0.754	0.783	9.528	9.525	3.04				
41	Butylbenzylphthalate	24.029	-3.9	305959	0.504	0.443	10.036	10.032	20.54	-0.0010	0.5264		0.9988
42	Benzo(a)anthracene	24.008	-4.0	650699	1.072	1.333	10.586	10.578	32.28	0.0028	1.1122		0.9999
43	Chrysene	21.791	-12.8	614002	1.012	1.161	10.620	10.613	12.30				
44	bis(2-Ethylhexyl)phthalate	24.234	-3.1	439413	0.724	0.680	10.616	10.612	15.30	-0.0047	0.7549		0.9998
45	Perylene-d12	40.000	0	876270	1	1	11.966	11.957	0				
46	Di-n-octylphthalate	27.244	9.0	728845	1.331	0.960	11.230	11.226	29.91	-0.0033	1.2261		0.9978
47	Benzo(b)fluoranthene	26.486	5.9	627061	1.145	1.081	11.596	11.586	4.32				
48	Benzo(k)fluoranthene	24.784	-0.9	604978	1.105	1.114	11.622	11.612	3.03				
49	Benzo(e)pyrene	25.233	0.9	615067	1.123	1.113	11.856	11.846	5.76				
50	Benzo(a)pyrene	26.038	4.2	584791	1.068	1.025	11.909	11.898	4.57				
51	Perylene	23.843	-4.6	640732	1.170	1.227	11.993	11.980	10.70				
52	Indeno(1,2,3-cd)pyrene	24.940	-0.2	655775	1.197	1.200	13.176	13.152	5.57				
53	Dibenzo(a,h)anthracene	24.818	-0.7	534379	0.976	0.983	13.199	13.171	5.02				
54	Benzo(g,h,i)perylene	23.966	-4.1	510179	0.932	0.972	13.539	13.508	5.84				





**Figure 7: TYPICAL SAMPLE RESULT SUMMARY**

METHOD 3550B/8270C SIM  
 SEMI VOLATILE ORGANICS BY GC/MS

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=====
Client      : XYZ, INC.                Date Collected: 06/01/11
Project     : CLEAN PROJECT           Date Received: 06/02/11
Batch No.   : 11F017                  Date Extracted: 06/06/11 14:50
Sample ID   : IR9SB01-2              Date Analyzed: 06/07/11 14:22
Lab Samp ID: F017-02                 Dilution Factor: 1
Lab File ID: RFH040                  Matrix          : SOIL
Ext Btch ID: SVF005S                 % Moisture     : 7.2
Calib. Ref.: REH072                  Instrument ID   : T-0E7
=====
  
```

PARAMETERS	RESULTS (ug/kg)	RL (ug/kg)	MDL (ug/kg)
ACENAPHTHENE	ND	11	2.7
ACENAPHTHYLENE	ND	11	2.7
ANTHRACENE	ND	11	2.7
BENZO(A)ANTHRACENE	ND	11	2.7
BENZO(A)PYRENE	ND	11	2.7
BENZO(B)FLUORANTHENE	ND	11	2.7
BENZO(K)FLUORANTHENE	ND	11	2.7
BENZO(G,H,I)PERYLENE	ND	11	2.7
CHRYSENE	ND	11	2.7
DIBENZ(A,H)ANTHRACENE	ND	11	2.7
FLUORANTHENE	ND	11	2.7
FLUORENE	ND	11	2.7
INDENO(1,2,3-CD)PYRENE	ND	11	2.7
NAPHTHALENE	ND	11	2.7
PHENANTHRENE	ND	11	2.7
PYRENE	ND	11	2.7
1,6,7-TRIMETHYLNAPHTHALENE	ND	11	2.7
1-METHYLNAPHTHALENE	ND	11	2.7
1-METHYLPHENANTHRENE	ND	11	2.7
2,6-DIMETHYLNAPHTHALENE	ND	11	2.7
2-METHYLNAPHTHALENE	ND	11	2.7
BENZO(E)PYRENE	3.5J	11	2.7
BIPHENYL	ND	11	2.7
PERYLENE	3.5J	11	2.7

SURROGATE PARAMETERS	RESULTS	SPK_AMT	% RECOVERY	QC LIMIT
TERPHENYL-D14	596	718.4	83.0	30-125
2-FLUOROBIPHENYL	495	718.4	68.9	45-105
NITROBENZENE-D5	480	718.4	66.8	35-100

**Figure 8: TYPICAL LCS/LCSD REPORT SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN PROJECT  
 BATCH NO.: 11F017  
 METHOD: METHOD 3550B/8270C SIM

MATRIX: SOIL % MOISTURE: NA  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: MBLK1S  
 LAB SAMP ID: SVF005SB SVF005SL SVF005SC  
 LAB FILE ID: RFH031 RFH032 RFH033  
 DATE EXTRACTED: 06/06/1114:50 06/06/1114:50 06/06/1114:50 DATE COLLECTED: NA  
 DATE ANALYZED: 06/07/1111:28 06/07/1111:47 06/07/1112:06 DATE RECEIVED: 06/06/11  
 PREP. BATCH: SVF005S SVF005S SVF005S  
 CALIB. REF: REH072 REH072 REH072

ACCESSION:

PARAMETER	BLNK RSLT (ug/kg)	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Acenaphthene	ND	1330	1110	83	1330	1100	82	1	45-110	30
Acenaphthylene	ND	1330	1180	88	1330	1160	87	2	45-105	30
Anthracene	ND	1330	1130	85	1330	1100	82	3	55-105	30
Benzo(a)anthracene	ND	1330	1220	92	1330	1200	90	2	50-110	30
Benzo(a)pyrene	ND	1330	1260	94	1330	1190	89	6	50-110	30
Benzo(b)fluoranthene	ND	1330	1280	96	1330	1240	93	3	45-115	30
Benzo(k)fluoranthene	ND	1330	1360	102	1330	1340	101	1	45-125	30
Benzo(g,h,i)perylene	ND	1330	1210	91	1330	1200	90	1	40-125	30
Chrysene	ND	1330	1160	87	1330	1140	86	2	55-110	20
Dibenz(a,h)anthracene	ND	1330	1220	92	1330	1210	91	1	40-125	30
Fluoranthene	ND	1330	1250	94	1330	1220	92	2	55-115	30
Fluorene	ND	1330	1190	89	1330	1170	88	1	50-110	30
Indeno(1,2,3-cd)pyrene	ND	1330	1220	91	1330	1210	91	1	40-120	30
Naphthalene	ND	1330	1020	76	1330	1010	76	1	40-105	30
Phenanthrene	ND	1330	1100	83	1330	1080	81	2	50-110	30
Pyrene	ND	1330	1210	91	1330	1180	89	2	45-125	30
1,6,7-Trimethylnaphthalene	ND	1330	1290	96	1330	1300	97	1	40-150	30
1-Methylnaphthalene	ND	1330	1060	79	1330	1040	78	2	40-150	30
1-Methylphenanthrene	ND	1330	1200	90	1330	1190	89	0	40-150	30
2,6-Dimethylnaphthalene	ND	1330	1140	86	1330	1160	87	1	40-150	30
2-Methylnaphthalene	ND	1330	1160	87	1330	1140	86	1	45-105	30
Benzo(e)pyrene	ND	1330	1290	96	1330	1290	97	1	40-150	30
Biphenyl	ND	1330	1130	85	1330	1130	85	0	40-150	30
Perylene	ND	1330	1210	90	1330	1210	91	1	40-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	BS RSLT (ug/kg)	BS % REC	SPIKE AMT (ug/kg)	BSD RSLT (ug/kg)	BSD % REC	QC LIMIT ( % )
Terphenyl-d14	667	618	93	667	651	98	30-125
2-Fluorobiphenyl	667	508	76	667	540	81	45-105
Nitrobenzene-d5	667	494	74	667	529	79	35-100

Figure 9: TYPICAL MS/MSD REPORT SUMMARY

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.  
 PROJECT: CLEAN PROJECT  
 BATCH NO.: 11F017  
 METHOD: METHOD 3550B/8270C SIM

MATRIX: SOIL % MOISTURE: 7.2  
 DILUTION FACTOR: 1 1 1  
 SAMPLE ID: IR9SB01-2  
 LAB SAMP ID: F017-02 F017-02M F017-02S  
 LAB FILE ID: RFH040 RFH036 RFH037  
 DATE EXTRACTED: 06/06/1114:50 06/06/1114:50 06/06/1114:50 DATE COLLECTED: 06/01/11  
 DATE ANALYZED: 06/07/1114:22 06/07/1113:03 06/07/1113:22 DATE RECEIVED: 06/02/11  
 PREP. BATCH: SVF005S SVF005S SVF005S  
 CALIB. REF: REH072 REH072 REH072

ACCESSION:

PARAMETER	SMPL RSLT (ug/kg)	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Acenaphthene	ND	1440	1210	84	1440	1010	70	18	45-110	30
Acenaphthylene	ND	1440	1270	89	1440	1060	74	18	45-105	30
Anthracene	ND	1440	1240	87	1440	1050	73	17	55-105	30
Benzo(a)anthracene	ND	1440	1350	94	1440	1140	79	17	50-110	30
Benzo(a)pyrene	ND	1440	1380	96	1440	1170	81	17	50-110	30
Benzo(b)fluoranthene	ND	1440	1460	102	1440	1210	84	18	45-115	30
Benzo(k)fluoranthene	ND	1440	1470	102	1440	1270	88	15	45-125	30
Benzo(g,h,i)perylene	ND	1440	1360	95	1440	1160	81	16	40-125	30
Chrysene	ND	1440	1280	89	1440	1080	75	17	55-110	30
Dibenz(a,h)anthracene	ND	1440	1380	96	1440	1180	82	16	40-125	30
Fluoranthene	ND	1440	1390	96	1440	1170	82	17	55-115	30
Fluorene	ND	1440	1320	92	1440	1110	77	17	50-110	30
Indeno(1,2,3-cd)pyrene	ND	1440	1380	96	1440	1180	82	16	40-120	30
Naphthalene	ND	1440	1010	70	1440	839	58	19	40-105	30
Phenanthrene	ND	1440	1240	86	1440	1040	72	17	50-110	30
Pyrene	ND	1440	1340	93	1440	1130	79	17	45-125	30
1,6,7-Trimethylnaphthalene	ND	1440	1340	93	1440	1160	81	14	40-150	30
1-Methylnaphthalene	ND	1440	1100	77	1440	912	63	19	40-150	30
1-Methylphenanthrene	ND	1440	1250	87	1440	1110	77	12	40-150	30
2,6-Dimethylnaphthalene	ND	1440	1160	81	1440	993	69	15	40-150	30
2-Methylnaphthalene	ND	1440	1190	83	1440	990	69	19	45-105	30
Benzo(e)pyrene	3.55J	1440	1350	94	1440	1190	83	12	40-150	30
Biphenyl	ND	1440	1200	83	1440	1010	71	17	40-150	30
Perylene	3.52J	1440	1260	88	1440	1110	77	12	40-150	30

SURROGATE PARAMETER	SPIKE AMT (ug/kg)	MS RSLT (ug/kg)	MS % REC	SPIKE AMT (ug/kg)	MSD RSLT (ug/kg)	MSD % REC	QC LIMIT ( % )
Terphenyl-d14	718	749	104	718	648	90	30-125
2-Fluorobiphenyl	718	582	81	718	497	69	45-105
Nitrobenzene-d5	718	548	76	718	469	65	35-100

Figure 10:

TYPICAL CASE NARRATIVE

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CASE NARRATIVE

Client : XYZ, INC.  
Project : CLEAN PROJECT  
SDG : 11F017

METHOD 3550B/8270C SIM  
SEMI VOLATILE ORGANICS BY GC/MS

A total of six (6) soil samples were received on 06/02/11 for PAH BY 8270C SIM analysis, Method 3550B/8270C SIM in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

Holding Time

Samples were analyzed within the prescribed holding time.

Instrument Performance and Calibration

Instrument tune check was performed prior to calibration. Instrument mass ratios were evaluated and results were within acceptance criteria. Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using secondary source (ICV). Continuing calibration (CCV) was carried on at a frequency required by the project. All project calibration requirements were satisfied. Refer to calibration summary forms for ICAL, ICV and CCV for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for SVF005SL/C were all within QC limits.

Matrix QC Sample

A set of MS/MSD was analyzed with the samples in this SDG. Percent recoveries for F017-02M/S were within project QC limits.

Surrogate

Surrogates were added on QC and field samples. Surrogate recoveries were within project QC limits.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameters.

## Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
DFTPP Tune Check	Prior to calibration (ICAL, ICV or CCV)	Refer to criteria listed in the method description (Table 4)	Retune instrument and verify		
Breakdown Check	Prior to calibration (ICAL, ICV or CCV)	Degradation $\leq$ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Clean the injection port and repeat breakdown check. If problem persist cut or replace column.		
ICAL: Multi-point calibration for all analytes	Initially; as needed	Min. RF: Refer to Table 6 %RSD $\leq$ 20% 1) for analytes with RSD $\leq$ 20% use RRFm 2) for analytes with RSD $>$ 20% and $r \geq 0.990$ use either inverse weighting factor or linear least squares with minimum of 6 ICAL points	Rule out outlier(s)/ bad injection/standard degradation, leak, etc. Correct as necessary otherwise repeat the ICAL		
ICV: Second-source calibration verification	After initial calibration	Min. RF: Refer to Table 6 All analytes within $\pm$ 30% of expected value	Rule out bad injection/standard degradation, leak, etc. Correct as necessary otherwise repeat the ICAL		
DCC: Calibration Check	Daily, before sample analysis and every 12 hours of analysis time	Min. RF: Refer to Table 6 All analytes $\leq$ 20% diff.	Rule out bad injection/standard degradation, leak, etc. Correct as necessary otherwise repeat the ICAL		
Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm$ 0.06 RRT units of the RRT	Correct the problem then reanalyze all samples analyzed since the last retention time check		
Internal Standard	Every sample, spiked sample, standard, and method blank	Retention time $\pm$ 30 seconds from retention time of the mid-point std. In the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning		
Method blank	One per preparation batch ( $\leq$ 20 samples per matrix)	In the absence of PSR apply No analytes detected $>$ $\frac{1}{2}$ LOQ	Rule out instrument contamination by re-analyzing the MB. If problem persist refer to PSR. In the absence of PSR, report NDs and results $>$ 10X of the MB concentration. Otherwise, cure contamination source, re-prep and re-analyze method blank and all associated samples.		
LCS	One LCS per preparation ( $\leq$ 20 samples per matrix)	In the absence of PSR default to EMAX QC Limits	Cure probable source of LCS failure, re-prep and reanalyze the LCS and all associated samples		
MS/MSD	One MS/MSD per every 20 project samples per matrix	In the absence of PSR default to EMAX QC Limits	Ensure that spike concentration and spike addition was accurate. If chromatogram exhibits matrix interference narrate observation in the case narrative.		
Surrogate spike	Every sample, spiked sample, standard, and method blank	In the absence of PSR default to EMAX QC Limits.	If non-compliant and no apparent matrix interference is observed, re-extract and analyze sample. Otherwise inform the client for further instruction.		
Comments: Project specific requirements (PSR) supersede EMAX QCP. In the absence of PSR, apply "J" flag to results between LOD and LOQ, "B" flag to results associated with MB contamination.			Reviewed By:		
			Date:		

Appendix 2:

DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY  
METHOD: SW 3550C / SW 8270SIM

Sample Prep SOP: EMAX-3550  
Analytical SOP: EMAX-8270SIM  
Conc Unit: µg/Kg  
Sample Amt(gm): 30  
Extract Volume (mL): 2

Instrument ID: E7  
Extraction date: 6/6/11 & 6/9/11  
Extracted by: J. Villena  
Analysis date: 6/7/11 & 6/9/11  
Analyzed by: D. Cheung

PARAMETER	RFH032	RFH033	RFH104	RFH105	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SVF005SL	SVF005SC	SVF012SL	SVF012SC							
Acenaphthene	1112	1100	1192	1126	1333	1132	85	41.0	4	40 - 130	PASSED
Acenaphthylene	1176	1156	1260	1184	1333	1194	90	45.5	4	50 - 130	PASSED
Anthracene	1135	1100	1208	1144	1333	1147	86	45.1	4	50 - 130	PASSED
Azobenzene	1200	1164	1324	1230	1333	1229	92	68.5	6	50 - 150	PASSED
Benzo(a)anthracene	1222	1200	1285	1369	1333	1269	95	75.8	6	50 - 130	PASSED
Benzo(a)pyrene	1258	1190	1356	1281	1333	1271	95	68.4	5	60 - 130	PASSED
Benzo(e)pyrene	1266	1294	1362	1309	1333	1313	98	34.5	3	60 - 130	PASSED
Benzo(b)fluoranthene	1275	1242	1437	1346	1333	1325	99	86.3	7	60 - 140	PASSED
Benzo(k)fluoranthene	1359	1344	1387	1270	1333	1340	100	49.9	4	60 - 140	PASSED
Benzo(g,h,i)perylene	1209	1197	1315	1279	1333	1250	94	56.5	5	60 - 130	PASSED
Bis(2-chloroethyl)ether	959	942	1024	971	1333	974	73	35.5	4	40 - 130	PASSED
Biphenyl	1129	1128	1207	1191	1333	1164	87	41.2	4	50 - 150	PASSED
Bis(2-Ethylhexyl)phthalate	1104	1083	1297	1347	1333	1208	91	133.8	11	50 - 150	PASSED
Butylbenzylphthalate	1084	1064	1283	1325	1333	1189	89	134.2	11	50 - 150	PASSED
Carbazole	1201	1179	1281	1268	1333	1232	92	49.8	4	70 - 130	PASSED
4-Chloro-3-methylphenol	1232	1220	1352	1267	1333	1268	95	59.7	5	40 - 130	PASSED
2-Chlorophenol	1036	1021	1100	1049	1333	1051	79	34.1	3	40 - 130	PASSED
Chrysene	1159	1141	1186	1265	1333	1188	89	54.5	5	50 - 140	PASSED
Dibenzo(a,h)anthracene	1221	1208	1364	1323	1333	1279	96	76.2	6	60 - 130	PASSED
2,4-Dichlorophenol	1156	1149	1235	1178	1333	1179	88	38.9	3	50 - 130	PASSED
Diethylphthalate	1252	1239	1363	1266	1333	1280	96	56.4	4	50 - 150	PASSED
2,6-Dimethylnaphthalene	1145	1159	1214	1181	1333	1175	88	30.1	3	50 - 130	PASSED
2,4-Dimethylphenol	1165	1146	1254	1194	1333	1190	89	47.2	4	50 - 130	PASSED
Dimethylphthalate	1202	1179	1294	1204	1333	1220	91	50.7	4	50 - 150	PASSED
Di-n-butylphthalate	1232	1204	1397	1363	1333	1299	97	95.2	7	50 - 150	PASSED
Di-n-octylphthalate	1098	1071	1401	1240	1333	1202	90	151.7	13	50 - 150	PASSED
Fluoranthene	1252	1223	1318	1314	1333	1277	96	46.8	4	60 - 140	PASSED
Fluorene	1186	1170	1270	1196	1333	1206	90	44.5	4	50 - 130	PASSED
Hexachlorobenzene	1219	1186	1288	1211	1333	1226	92	43.8	4	50 - 130	PASSED
Indeno(1,2,3-cd)pyrene	1220	1208	1368	1328	1333	1281	96	79.5	6	60 - 130	PASSED
1-Methylnaphthalene	1059	1042	1123	1066	1333	1072	80	34.9	3	50 - 130	PASSED
2-Methylnaphthalene	1158	1142	1229	1166	1333	1174	88	38.1	3	50 - 130	PASSED
1-Methylphenanthrene	1198	1193	1252	1242	1333	1221	92	30.1	2	70 - 130	PASSED
Naphthalene	1019	1009	1076	1024	1333	1032	77	30.2	3	50 - 130	PASSED
N-Nitrosodimethylamine	1054	1019	1103	1057	1333	1058	79	34.3	3	50 - 150	PASSED
N-Nitroso-di-n-propylamine	1113	1097	1228	1157	1333	1149	86	58.5	5	40 - 130	PASSED
Perylene	1206	1214	1155	1114	1333	1172	88	46.8	4	60 - 130	PASSED
Phenanthrene	1104	1080	1171	1112	1333	1117	84	38.3	3	50 - 130	PASSED
Pyrene	1207	1183	1261	1266	1333	1229	92	41.1	3	50 - 130	PASSED
Pentachlorophenol	1139	1131	1209	1184	1333	1166	87	37.2	3	50 - 150	PASSED
Phenol	1006	988	1077	1038	1333	1027	77	39.0	4	40 - 130	PASSED

**Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY**

**DEMONSTRATION OF CAPABILITY  
METHOD: SW 3550C / SW 8270SIM**

Sample Prep SOP: EMAX-3550  
Analytical SOP: EMAX-8270SIM  
Conc Unit: µg/Kg  
Sample Amt(gm): 30  
Extract Volume (mL): 2

Instrument ID: E7  
Extraction date: 6/6/11 & 6/9/11  
Extracted by: J. Villena  
Analysis date: 6/7/11 & 6/9/11  
Analyzed by: D. Cheung

PARAMETER	RFH032	RFH033	RFH104	RFH105	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SVF005SL	SVF005SC	SVF012SL	SVF012SC							
2,4,6-Trichlorophenol	1288	1278	1391	1309	1333	1317	99	51.4	4	50 - 130	PASSED
2,4,5-Trichlorophenol	1303	1289	1380	1288	1333	1315	99	43.8	3	50 - 130	PASSED
2,3,5-Trimethylnaphthalene	1287	1295	1373	1326	1333	1320	99	39.2	3	50 - 150	PASSED
2-Fluorophenol	1408	1483	1526	1514	2000	1483	74	53.3	4	30 - 130	PASSED
Phenol-d5	1459	1543	1606	1611	2000	1555	78	70.6	5	30 - 130	PASSED
Nitrobenzene-d5	494	529	545	541	667	527	79	23.2	4	40 - 130	PASSED
2-Fluorobiphenyl	508	540	547	542	667	534	80	17.5	3	40 - 130	PASSED
2,4,6-Tribromophenol	1832	1910	1988	1933	2000	1915	96	64.8	3	50 - 130	PASSED
Terphenyl-d14	619	651	657	694	667	655	98	30.8	5	50 - 130	PASSED



8270SIMFA:

ANALYTICAL RUN LOG



ANALYSIS LOG FOR SEMIVOLATILES

SOP  EMAX-8270 Rev. No. \_\_  EMAX-8270SIM Rev. No. \_\_  EMAX-CLPSVOA  EMAX-M8270SIM Rev. No. \_\_  EMAX-625 Rev. No. \_\_

Book #AE7 -006

Method File:		Tune File:		Start Date/Time:			End Date/Time:		
Preparative Batch	Data File Name	Run ID	DF	Matrix		Notes	Instrument No:		E7
				S	W				
							INITIAL CALIBRATION REFERENCE		
							Date		
							ICAL ID		
							Standards		
							Name	ID	Conc. (mg/L)
							DFTPP		
							INT. STD.		
							ICV		
							DCC		
							BENZIDINE		
							APP 9		
							APP 9 ADD		
							Solvent	ID	
							CH <sub>2</sub> Cl <sub>2</sub>		
							DATA FILE		
							Electronic Data Archival		
							Location	Date	
							HPCHEM_SVOA/TOE7		
							Comments: _____		
							Analyzed By: _____		
							Date Disposed: _____		
							Disposed By: _____		
							This page is checked during data review.		

ANALYTICAL BATCH: \_\_\_\_\_





## Standard Operating Procedure

### Instrumental Analysis of Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) BY HRGC-HRMS (EPA METHOD 8290)

#### **STATEMENT OF PURPOSE**

This procedure describes the proper way to extract and cleanup samples from solid, tissue, and aqueous matrices and analyze them for PCDD (Dioxins) and PCDF (Furans) using high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

#### **INSTRUCTIONS**

##### **1.0 Scope and Application**

- 1.1 This method describes the part-per-quadrillion (ppq) instrumental analysis of PCDD (Dioxins) and PCDF (Furans) in extracts from solid, tissue and aqueous matrices using high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). This method also describes the required extraction and cleanup steps for all matrices prior to analysis.
- 1.2 The following compounds can be determined by this method:

Compound	CAS No.
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-39-4
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0

- 1.3 The quantitation range of 2,3,7,8-TCDD for 1 liter water sample (final vol. 50 $\mu$ L) is 5 – 10,000 ppq (pg/L). The quantitation range of 2,3,7,8-TCDD for 10g soil/tissue (final vol. 50 $\mu$ L) is 0.5 – 1,000ppq (pg/g). Reporting limits are based upon the lowest calibration point and the dilution factor, however Practical Quantitation Limits (PQL) are dependent on the potential interferences caused by the sample matrix.
- 1.4 The Toxicity Equivalent Quotient (TEQ) using 2,3,7,8 TCDD may also be determined by this method.



- 1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. If an individual project has its own QAPP with client specific requirements that are different than the SOP, the QAPP overrides the SOP. This information will be specified in the comment section of the ARF.
- 1.6 Precautions must be taken to exposure to materials known or believed to contain PCDDs or PCDFs. Many of these compounds are extremely toxic and it is the responsibility of laboratory personnel to practice safe laboratory procedures.

**2.0 Method Summary**

- 2.1 Samples are spike with internal standards then extracted and cleaned up using a variety of procedures. Extraction processes may include Soxhlet or separatory funnel shake. Clean up procedures may include acid/base shake, acid/base silica gel column, and carbon column.
- 2.2 C<sub>13</sub> labeled recovery standards are added to each sample extract prior to instrument analysis. The analytes are separated by HRGC using a Restek DB-5 column and detected by HRMS.
- 2.3 An analyte is identified in a sample by comparing the RT and ion abundance ratios (of the two most abundant m/z) to the standard RT and theoretical ion abundance ratios.
- 2.4 Quantitation is achieved based on a minimum five-point calibration curve, using the internal standard quantitation technique.

**3.0 Detection Limits**

The following list shows the various sample types and the 2,3,7,8-TCDD-based method calibration limits (MCLs) that are cover in this procedure.

	Water	Soil	Fish Tissue
Lower MCL	5 pg/L	0.5 pg/g	0.5pg/g
Upper MCL	10000 pg/L	1000 pg/g	1000 pg/g
Weight / Volume	1000 mL	10g	10g
IS spiking levels	5000pg/L	500pg/g	500pg/g
Final volume	50µl	50µl	50µl

**4.0 Definitions**

Calibration standard - A solution prepared from the primary dilution standard solution or stock standard solution and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration.

Detection Limit (DL) - The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. (NELAC) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Extracted Ion Current Profile (EICP)—The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number.



**Field Reagent Blank** - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

**Instrument blank (Blk)** - An aliquot of reagent water or other blank matrix to demonstrate that the instrument is not contributing contaminants to the samples.

**Internal Standard (IS)** - A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

**Instrument Performance Check (IPC)** - A solution of one or more compounds (analytes, surrogate, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

**Laboratory control spike (LCS)** - An aliquot of reagent water or other matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

**Laboratory Reagent Blank** - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

**Limit of Detection** - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%)

**Limit of Quantitation** - The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. This also equates with the term Practical Quantitation Limit (PQL).

**Matrix** - A surrounding substance within which something originates, develops, or is contained, such as: drinking water, saline/estuarine water, aqueous substance other than drinking water or saline/estuarine water, non-aqueous liquid, biological tissue, solids, soils, chemical waste, and air.

**Matrix duplicate (MD)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix sample and matrix sample duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.



**Matrix spike (MS)** - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.

**Matrix spike duplicate (MSD)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix spike and matrix spike duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**Method blank** - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

**Method Calibration Limit, Lower (LCML)** – The low standard on the calibration method.

**Method Calibration Limit, Upper (UCML)** – The high standard on the calibration method.

**Method detection limit (MDL)** - The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte., as described in 40 CFR Part 136, Appendix B, 1 July 1995 edition.

**Practical quantitation limit** - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The practical quantitation limit is generally three to ten times greater than the method detection limit.

**Primary Dilution Standard** - A solution of several analytes prepared in the laboratory from stock solution and diluted as needed to prepare calibrations solutions and other needed analyte solutions.

**Quality Control Sample (QCS)** - A solution of method analytes of known concentrations which is used to fortify an aliquot of LCS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

**Sample Duplicate (DUP1/DUP2)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analytes of DUP1/DUP2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**Stock Standard Solution** - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials purchased from a reputable commercial source.

**Surrogate** - A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is



measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample.

### 5.0 Interferences and Potential Problems

- 5.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Baking of glassware in a kiln or furnace at 450 - 500 °C may be necessary to remove contaminants.
- 5.2 All materials used in the analysis must be demonstrated to be free from interferences by running reference matrix method blanks before each sample batch.
- 5.3 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the target analytes. The most frequently encountered interferences are polychlorinated biphenyls. Because very low levels of Dioxins and Furans are measured by this method, the elimination of interferences is essential. The cleanup steps given in the extraction SOPs can be used to reduce or eliminate these interferences and thereby permit reliable determination of the analytes at low levels.
- 5.4 In order to prevent contamination of the calibration solutions, the solutions must be prepared in an area free from contamination using glassware free from contamination.
- 5.5 If the laboratory air is a potential source of contamination, samples, reagents, glassware, and other materials should be dried in a fume hood or other area free from contamination.

### 6.0 Health and Safety

- 6.1 Lab coats and gloves are to be used at all times. Follow all safety procedures as describes in the SOPs for samples suspected of containing biological hazards.
- 6.2 2,3,7,8-TCDD has been classified as a known human or mammalian carcinogen and teratogen. On the basis of the available toxicological and physical properties of the Dioxins and Furans, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks. Standards should be prepared using gloves, goggles and lab coats under a fume hood. Soil sample homogenization should be performed under a hood to reduce inhalation of dust particles.
- 6.3 The pure standards and samples suspected to contain these compounds are handled using essentially the same techniques employed in handling radioactive or infectious materials. Well-ventilated, controlled access laboratories are required. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or Labor, many of which have an industrial health service.
- 6.4 Protective Equipment
  - 6.4.1 Handle standards and samples under a hood to minimize contamination of the laboratory.
  - 6.4.2 Always wear safety glasses and throw away any plastic gloves, apron, or lab coat after each use.
  - 6.4.3 PVC gloves should never be used.



- 6.5 Training
  - 6.5.1 Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.
- 6.6 Personal Hygiene
  - 6.6.1 Thoroughly wash hands and forearms after each manipulation and before breaks.
- 6.7 Confinement - Dioxin and furan extractions require an isolated work area posted with signs.
- 6.8 Waste
  - 6.8.1 Try to minimize any contaminated waste.
  - 6.8.2 Use plastic bag liners in waste cans.
  - 6.8.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with UV light at a wavelength less than 290nm for several days. Dispose when the 2,3,7,8-TCDD/TCDF congeners can no longer be detected.
- 6.9 Decontamination
  - 6.9.1 If exposed to skin, apply a mild soap with plenty of scrubbing action.
  - 6.9.2 On glassware, tools, and other surfaces, use a solvent such as Methylene chloride. First rinse with Methylene chloride then wash with a detergent and water. Follow with a series of four rinses: 1) methanol, 2) 50% Methylene chloride 50% Hexane, 3) Witches Brew [containing equal amounts of toluene, hexane, acetone, and Methylene chloride], 4) Methylene chloride.
- 6.10 Laundry
  - 6.10.1 Contaminated clothing need to be kept away from other clothing in a designated container labeled under "Disposal of Hazardous Wastes."
  - 6.10.2 Contaminated clothing to be laundered should be collected in plastic bags to minimize any direct contact. Clothing could be de-contaminated by running them through a full laundry cycle. An empty laundry cycle should be run afterwards to clean up any contamination.
- 6.11 Inhalation
  - 6.11.1 It is good practice to provide good ventilation and to work under a hood to minimize any airborne contamination.
  - 6.11.2 Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion.
  - 6.11.3 Masks fitted with charcoal filters are recommended to prevent inhalation of dust.
- 6.12 Accidents
  - 6.12.1 Remove any contaminated clothing immediately
  - 6.12.2 Wash exposed skin vigorously and repeatedly until medical attention is obtained.
- 6.13 Employee Health Monitoring
  - 6.13.1 A urinary porphyrin profile may be conducted annually as needed in order to monitor the toxic body burden for employees who work directly with Dioxin/Furan sample extraction.
  - 6.13.2 Porphyrins are enzymes involved in the formation of the blood component, heme, measured in urine. Heme is essential for the proper function of many proteins including oxygen transport, energy production, and detoxification. Proper porphyrin production is essential for our body's capacity to detoxify toxins.
  - 6.13.3 Any disturbance in the Heme chemical pathway tends to cause rapid and relatively large accumulations of porphyrins. These enzymes are widely



distributed in human tissues, and are highly sensitive to the presence of various toxins (such as metals, dioxins or pesticides), creating the large accumulation of porphyrins in the urine<sup>2</sup>.

- 6.13.4 The urine test kits may be ordered through Direct Labs 1-800-308-0000 ([www.directlabs.com](http://www.directlabs.com)). The kit can then be used at home and mailed to Metamatrix for porphyrin profiling ([www.metamatrix.com](http://www.metamatrix.com)). The necessary paper work and mailing instructions are provided by Metamatrix in the kit. The results are recorded in a logbook.
- 6.13.5 If the porphyrin profile exhibits elevated enzyme levels corresponding to possible toxic build-up in the body, then further blood analysis may be done.
- 6.14 Wipe Tests This procedure is used for periodic evaluation of potential contamination of dioxins and furans in the working environment. It is recommended to perform a wipe test at a minimum when there is evidence of contamination in the method blanks.
- 6.14.1 Collecting wipe samples
- 6.14.1.1 Using stainless steel forceps and glass fiber paper saturated with distilled acetone, wipe down surface areas of two inches by one foot.
  - 6.14.1.2 Combine the wipes in a glass jar containing 200mL distilled acetone.
  - 6.14.1.3 For the control, place an equal number of unused wipers in a glass jar containing 200mL distilled acetone
  - 6.14.1.4 Spike each composite sample with 20 $\mu$ L of sample fortification solution.
- 6.14.2 Extraction
- 6.14.2.1 Shake jar for 20 minutes and transfer extract to a Rotovap flask.
  - 6.14.2.2 Using a Rotovap, concentrate down to 1.0 mL.
  - 6.14.2.3 Using hexane, transfer extract to a centrifuge tube and concentrate down to 300 $\mu$ L.
  - 6.14.2.4 Transfer to injection vial and using nitrogen, concentrate down to 30 $\mu$ L.
  - 6.14.2.5 Rinse centrifuge tube twice with 300 $\mu$ L of hexane and transfer to injection vial.
  - 6.14.2.6 Solvent transfer extract to about 30 $\mu$ L of nonane.
  - 6.14.2.7 Spike sample with 20 $\mu$ L of recovery standard.
- 6.14.3 Calculations
- 6.14.3.1 Report as ng/wipe (WTE).
  - 6.14.3.2 A lower limit of 10pg/WTE is expected for 2,3,7,8-TCDD.
  - 6.14.3.3 A positive response for 2,3,7,8-TCDD is considered any hit at or above 3pg/WTE.
  - 6.14.3.4 The multiplication factor for TCDF/PeCDD/PeCDF = 1.
  - 6.14.3.5 The multiplication factor for HxCDD/HxCDF/HpCDD/HpCDF = 2.5.
  - 6.14.3.6 The multiplication factor for OCDD/OCDF = 5.  
For example, in the case of HxCDD, the lower MCL is 10 X 2.5 = 25pg/WTE and the positive response for the blank would be 3 X 2.5 = 7.5pg.

## 7.0 Sample Preservation, Containers, Handling and Storage

- 7.1 Containers used to collect samples for the determination of Dioxin and Furan compounds are purchased pre-cleaned. The sample containers for solids are wide mouth, amber glass, 500mL minimum containers with Teflon lined screw caps. The sample containers for waters are 1 liter amber glass bottles with Teflon lined screw caps.



Filletted fish tissue samples may be collected in aluminum foil and kept frozen until receipt by the laboratory.

- 7.2 All samples will be taken and held at a temperature of  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  in the dark until delivery to the laboratory. Water and solid samples are then placed into a refrigerator that is kept at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  until extraction. Tissue samples are to be stored frozen by the laboratory until extraction.
- 7.3 For solids and aqueous samples, extraction hold time is 30 days from date of collection, and analysis hold time is 45 days from date of extraction.
- 7.4 For tissue samples, extraction hold time is 30 days from date of collection, and analysis hold time is 45 days from date of collection.

### 8.0 Quality Control

- 8.1 Each analyst must have an initial demonstration of competency study (IDC) for solid and aqueous matrices, by generating data of acceptable accuracy and precision for target analytes using four replicate spikes that have undergone the entire preparation, extraction, and cleanup procedures outlined in the method and respective APPL SOPs. The laboratory must also repeat the following operations whenever new staff is trained. See Table 1 of this SOP for recovery and %RSD limits for the IDC.
- 8.2 Sample quality control for preparation and analysis include the analysis of a method blank, a matrix spike/matrix spike duplicate, and laboratory control sample in each analytical batch of 20 samples, and the addition of labeled internal standards and labeled C13 surrogates to each sample and QC sample.
- 8.3 The method blank must be shown to contain no analytes of interest above  $\frac{1}{2}$  the RL. If sample volume allows, samples with hits of analytes detected above  $\frac{1}{2}$  the RL in the method blank must be reanalyzed. Acceptance criteria for DoD clients: No analytes detected at  $\geq \frac{1}{2}$  LOQ.
- 8.4 Corrective Action: If there is a detection above the quantitation limit (or  $> \frac{1}{2}$  LOQ for DoD) in the method blank the entire batch associated with the blank will be re-extracted and reanalyzed except when the sample analysis resulted in a non-detect. If not enough sample volume exists for a re-extraction the sample will be qualified with a 'B' with the flag 'compound found in the associated blank.
- 8.5 The laboratory control sample will be included with each batch of 20 samples or less. The LCS consists of an aliquot of control matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate. See Table 1 in this SOP for compounds, spike levels and spike acceptance criteria. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
- 8.6 The matrix spike and matrix spike duplicate will be included with each sample batch as per client. The client is to determine which sample is designated with MS/MSD. The %RPD limit for the MS/MSD is  $\leq 20\%$ . If the criteria is not met notify the project manager who will examine the DQOs and contact the client.
- 8.7 Any native hits associated in the sample and sample duplicate, if applicable, must have at %RPD limit of  $\leq 25\%$ . For DoD clients the acceptance limits for sample and sample duplicate must be  $\leq 20\%$ .
- 8.8 If the recoveries of the LCS, MS/MSD, or sample duplicate are not within limits, the following are required:
  - 8.8.1 Confirm that there are no errors in calculations or surrogate solutions.
  - 8.8.2 Instrument performance should also be checked for problems.



- 8.8.3 Examine chromatograms for interfering peaks and for integrated areas.
- 8.8.4 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- 8.8.5 Re-extract and reanalyze the sample if none of the above is the problem.
- 8.9 The EPA 8290 method does not have an MDL requirement; however APPL Inc. will perform an initial MDL study for both soil and water extraction methods and cleanup procedures and then quarterly MDL checks according to SOP QC018. The study samples will not be screened for J-values between the MDL and the PQL, since the method requires screening to the dynamic LOD/EDL or EMPC values generated by the quantitation software. These values are significantly lower than the PQL.
- 8.10 Deviations: Any activity not performed in accordance with laboratory procedures or Quality Assurance Project Plans is considered a deviation from plan. All deviations from plan will be documented as to the extent of, and reason for, the deviation.
- 8.11 Corrective Action: Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria will be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken is documented on a Nonconformance Work Report (NWR) and/or a Corrective Action Report (CAR).
- 8.12 Data Reporting Criteria: Data is obtained from the primary column. If all the isomers are resolved within 25% using the DB-5 column, then the DB-225 confirmation column is not needed.

### 9.0 Equipment/Apparatus

- 9.1 Gas chromatograph—Must have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and must meet all of the performance specifications in the method.
- 9.2 GC column
  - 9.2.1 The suggested primary column is the 60m DB-5 from Restek, and the suggested confirmation column (for resolving 2,3,7,8-TCDF from the individual isomers) is 30m DB-225. If the isomers listed in the method (2,3,7,8-TCDF from 2,3,4,7-TCDF and 1,2,3,9-TCDF) are resolved by 25% on the DB-5 column, then the confirmation column analysis is not necessary for 2,3,7,8-TCDF.
  - 9.2.2 The column must meet the specifications listed in this SOP for retention time and resolution of peaks.
- 9.3 Mass spectrometer - electron impact ionization, must be capable of selectively monitoring ions 304.9824 and 380.9760 at minimum high resolution (10,000) during a period less than 1.0 second.
- 9.4 GC/MS interface - The mass spectrometer (MS) must be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.
- 9.5 Data system—Capable of collecting, recording, storing, and processing MS data (MassLynx Software). Data acquisition—The signal at each exact m/z must be collected repetitively throughout the monitoring period and stored on a mass storage device.
- 9.6 Laboratory Fume Hood.
- 9.7 Analytical balance capable of weight  $\pm 0.1$  mg.
- 9.8 Extraction apparatus



- 9.8.1 Soxhlet extractor apparatus
- 9.8.2 Soxhlet 50mm ID, 200mL capacity with 500mL flask
- 9.8.3 Heating mantle
- 9.8.4 Chiller
- 9.8.5 Liquid/liquid 2000mL separatory funnels with fluoropolymer stopcocks
- 9.8.6 125mL Separatory funnel (for soil-back extraction)
- 9.9 Glassware (Class A)
  - 9.9.1 500mL flat bottom boiling flasks
  - 9.9.2 1-L graduated cylinder (Class A)
  - 9.9.3 Beakers 400 to 500mL
  - 9.9.4 Conical glass centrifuge tube (15mL)
  - 9.9.5 Erlenmeyer flasks (250mL)
  - 9.9.6 Glass funnel 125 to 250mL
  - 9.9.7 Glass wool, extracted with Methylene chloride, dried and stored in a clean glass jar.
  - 9.9.8 Glass-fiber filter paper
  - 9.9.9 Pipettes, disposable, Pasteur, 150mm long X 5mm ID
  - 9.9.10 Pipettes, disposable, serological, 25mL
  - 9.9.11 Sample vials – 2mL amber glass
  - 9.9.12 Sample vials – 0.3mL conical glass
  - 9.9.13 Class A volumetric syringes-10 $\mu$ L, 100 $\mu$ L, 500 $\mu$ L, and 1.0mL for standard and spike preparation.
- 9.10 Miscellaneous Equipment
  - 9.10.1 Blender
  - 9.10.2 Desiccator
  - 9.10.3 Laboratory oven capable of sustaining temperatures up to 400° C
  - 9.10.4 Glass-fiber thimble
  - 9.10.5 Mortar and Pestle
  - 9.10.6 Aluminum foil
  - 9.10.7 Stainless steel spoons and spatulas
- 9.11 Concentration apparatus
  - 9.11.1 Rotary evaporator
    - 9.11.1.1 Vacuum for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge.
    - 9.11.1.2 A recirculating water pump and chiller.
    - 9.11.1.3 Adjustable water bath
- 9.12 Nitrogen blowdown apparatus.

### 10.0 Reagents and Standards

- 10.1 Reagent or pesticide grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All reagent and chemical lots will be documented properly for traceability. Reference standards must be calibrated by a body that can provide ILAC-signatory (MRA) traceability.
- 10.2 Store the standard solutions (stock, calibration and internal standards) at room temperature in Teflon-sealed containers in the dark. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem. It is recommended that the laboratory purchase dilute standard solutions of the



analytes in this Method. If primary solutions are prepared, they must be prepared in a hood.

- 10.3 Organic-free reagent water: All references to water in this method refer to organic-free reagent water.
- 10.4 Stock standard solutions may be purchased as individual manufacturer certified solutions. The certified solutions must be accompanied by a certificate of analysis that states balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSS Z-540-1 and ISO 9001. Reference standards must be calibrated by a body that can provide ILAC-signatory (MRA) traceability.
- 10.4.1 Calibration Standards- may be purchased from a manufacturer such as Cambridge Isotope Laboratories in five separate solutions in nonane, as listed in Table 1 of this SOP. The calibration standards should include the seventeen unlabeled Dioxins and Furans, as well as the nine C13-labeled internal standards and the two C13-labeled recovery standards. The CS-3 standard is used for calibration verification (CCV).
- 10.4.2 Performance check solution- may be purchased in nonane solvent from a manufacturer such as Cambridge Isotope Laboratories to include the compounds listed in Table 2 of this SOP.
- 10.4.3 Internal Standards- may be purchased from a manufacturer such as Cambridge Isotope Laboratories to include the compounds listed in Table 3 of this SOP. This mix is prepared in nonane and added to each sample, blank and spike prior to extraction.
- 10.4.4 Recovery Standards- may be purchased from a manufacturer such as Cambridge Isotope Laboratories to include the compounds listed in Table 3 of this SOP. This mix is prepared in nonane and added to each sample, blank and spike at the final concentration step of the extraction procedure.
- 10.4.5 Spike Mix- may be purchased from a manufacturer such as Cambridge Isotope Laboratories to include the compounds listed in Table 4 of this SOP. This mix is prepared in nonane and spiked to each LCS and MS/ MSD prior to extraction.
- 10.5 Reagents.
- 10.5.1 Organic free water
- 10.5.2 Quartz sand (or Ottawa Sand)
- 10.5.3 Concentrated Sulfuric acid – reagent grade
- 10.5.4 20% Potassium Hydroxide solution - (20g KOH dissolved into 100mL DI Water)
- 10.5.5 5% Sodium Chloride solution – (5g NaCl dissolved into 100mL of DI Water)
- 10.5.6 Methylene chloride – reagent grade
- 10.5.7 Nonane – reagent grade
- 10.5.8 Toluene – reagent grade
- 10.5.9 Hexane – reagent grade
- 10.5.10 Acetone – reagent grade
- 10.5.11 Methanol – reagent grade
- 10.5.12 Cyclohexane – reagent grade
- 10.5.13 Activated Silica Gel-100 mesh (Soxhlet overnight with Methylene chloride then bake overnight @ 180°C: cooled in Desiccator and stored in glass jar with Teflon-screw cap)
- 10.5.14 Acidic Silica Gel (100g activated silica gel + 44g concentrated H<sub>2</sub>SO<sub>4</sub> mixed well and stored in glass jar with Teflon-screw cap)



- 10.5.15 Basic Silica Gel (100g activated silica gel + 30g of 1N NaOH mixed well and stored in glass jar with Teflon screw cap)
- 10.5.16 Celite 545 (stored in a sealed container at room temperature)
- 10.5.17 Active carbon AX-21 (prewashed with methanol and dried in vacuum at 110°C. Store in a glass bottle sealed with a Teflon lined screw cap.
- 10.5.18 Activated Anhydrous Sodium Sulfate (Soxhlet overnight with Methylene chloride then bake for four hours @ 400°C: cooled in Desiccator and stored in glass jar with Teflon-screw cap)

### 11.0 Calibration and Standardization

Please refer to the Procedure section on this SOP for any calibration and standardization instructions.

### 12.0 Procedure

#### 12.1 Aqueous Samples

##### 12.1.1 Dioxin/Furan Separatory Funnel Extraction (EPA Method 8290)

- 12.1.1.1 Allow time for sample to reach room temperature.
- 12.1.1.2 Mark the water meniscus of the sample bottle to later determine the exact sample volume.
- 12.1.1.3 Spike the samples, blanks, LCS and MS/MSD with the acetone diluted surrogate solution. Spike the LCS and MS/MSD with spike mix.

**NOTE:** The addition of spike and surrogate will be witnessed by a second person and will be documented on the extraction sheet.

- 12.1.1.4 For samples with >1% solids, filter through a 0.45µm glass fiber filter that has been rinsed with toluene. If there is too much solids to filter, then centrifuge sample, decant and then filter the aqueous phase.
- 12.1.1.5 Combine the solids from the filter paper and the centrifuge contents and proceed to the extraction process for soils/sediment for this sample portion.
- 12.1.1.6 Pour the aqueous filtrate into a 2L separatory funnel. Rinse out the sample bottle with 60mL Methylene chloride and add the liquid to the separatory funnel. Shake the funnel for two minutes with periodic venting.
- 12.1.1.7 Allow the organic layer to separate from the water phase. Making sure the emulsion interface between layers is not more than one third the volume of the solvent layer.
- 12.1.1.8 Pass the Methylene chloride through a filter funnel packed with a glass wool plug and 5g anhydrous sodium sulfate into a Rotovap flask.
- 12.1.1.9 Repeat the extraction twice with fresh 60mL portions of Methylene chloride. After the third extraction, rinse the sodium sulfate filter funnel with an additional 30mL Methylene chloride to ensure quantitative transfer.
- 12.1.1.10 Concentrate the extract in a rotary evaporator (35°C water bath), to a volume of 5mL. Allow it to cool.



- 12.1.1.11 Add 50mL hexane and (if applicable) the concentrate obtained from the solid soil extraction and concentrate down to 5mL.
  - 12.1.1.12 Transfer the concentrate to a 125mL separatory funnel.
  - 12.1.1.13 Rinse the flask and the lower joint with two 15mL portions of hexane and combine the rinses with the extract to ensure quantitative transfer.
  - 12.1.1.14 Determine the original sample volume of the sample bottle by filling the sample bottle to the mark and determining the volume by using a 1000mL graduated cylinder. Record the sample volume to the nearest 5mL.
  - 12.1.1.15 Proceed to cleanup stage.
- 12.2 Soils and Sediment Samples
- 12.2.1 Allow time for sample to reach room temperature.
  - 12.2.2 Add 10g of anhydrous powdered sodium sulfate to 10g of soil (dry weight) and mix thoroughly.
  - 12.2.3 Place mixture in Soxhlet apparatus on top of a glass wool plug or Soxhlet thimble and add 250mL of Methylene chloride to the boiling flask.
  - 12.2.4 Spike the samples, blanks, LCS and MS/MSD with the acetone diluted surrogate solution. Spike the LCS and MS/MSD with spike mix.
- NOTE:** The addition of spike and surrogate will be witnessed by a second person and will be documented on the extraction sheet.
- 12.2.5 Reflux for 16 hours. The solvent must cycle completely through the system five times per hour.
  - 12.2.6 Cool and filter through a glass fiber filter into a 500mL round bottom flask.
  - 12.2.7 Rinse filter with 10mL of Methylene chloride and using a Rotovap, concentrate to near dryness at 40°C. Solvent transfer to hexane. Allow to cool.
  - 12.2.8 Transfer the residue to a 125mL separatory funnel with 15mL of hexane and rinse flask with two additional 15mL portions of hexane.
  - 12.2.9 Proceed to cleanup stage.
- 12.3 Fish Tissue / Paper Pulp Samples
- 12.3.1 For fish tissue samples, homogenize at least 20 grams of frozen fish tissue using a stainless steel meat grinder with a 3 to 5 mm hole size inner plate. The client may specify which part of the fish is to be tested (i.e. skin, edible meat, vital organs or the entire fish).
  - 12.3.2 Weigh two 10g (dry weight) aliquots of homogenized fish tissue sample into clean glass beakers. One aliquot will be used for dioxin analysis and the other for lipid determination.
  - 12.3.3 Prepare glass beakers for the method blank and the lab control spike using 1-2 grams of vegetable or corn oil. One blank and LCS per 20 samples or less. If the client requests a matrix spike/matrix spike duplicate, then weigh 10g (dry weight) of tissue into two separate glass beakers.
  - 12.3.4 Add 60g anhydrous sodium sulfate to each of the beakers and mix thoroughly.
  - 12.3.5 Add 250mL of Methylene chloride or hexane/Methylene chloride (1:1 v:v) to the Soxhlet apparatus.
  - 12.3.6 Spike the samples, blanks, LCS and MS/MSD with the acetone diluted surrogate solution. Spike the LCS and MS/MSD with spike mix.



**NOTE:** The addition of spike and surrogate will be witnessed by a second person and will be documented on the extraction sheet.

- 12.3.7 Transfer sample from glass beakers to Soxhlet thimbles.
- 12.3.8 Reflux for 16 hours. The solvent must cycle completely through the system five times per hour.
- 12.3.9 For partially dewatered paper pulp samples, follow the same steps using different amounts: 10g sample, 30g of anhydrous sodium sulfate and 250mL of toluene.
- 12.3.10 Concentrate the extract in a Rotovap to a volume of 10mL. Allow to cool for 5 min. Perform a solvent transfer from the Methylene chloride to hexane. Make sure all the Methylene chloride have been completely removed before proceeding with the next step.
- 12.3.11 Decant the contents of the flask into a 125mL separatory funnel.
- 12.3.12 Rinse the flask with two additional 15mL portions of hexane and add the rinses to the funnel.
- 12.3.13 Proceed to cleanup stage.
- 12.3.14 **Lipid Content Determination**  
Tare a 250mL round bottom flask and add the second aliquot fish tissue extract. Rinse the flask with two additional 5mL portions of hexane and add the rinses to the flask. Concentrate fish tissue on a rotary evaporator until a constant weight is achieved.

$$\text{Percent lipid} = \frac{100 \times \text{weight of concentrate}}{10}$$

#### 12.4 Cleanup Stage

##### 12.4.1 Acid/Base Partition

- 12.4.1.1 Add 40mL of the concentrated  $\text{H}_2\text{SO}_4$  to the hexane extract and shake for 2min with periodic venting and discard the aqueous layer. Repeat the "acid washing" until no color is visible in the aqueous layer for a maximum of 4 washings.
- 12.4.1.2 Add 40mL of NaCl solution and shake for 2min with periodic venting and discard the aqueous layer.
- 12.4.1.3 Add 40mL of 20% potassium hydroxide. Shake for 2min and discard the aqueous layer. Repeat the "base washing" until no color is visible in the aqueous layer for a maximum of 4 washings.
- 12.4.1.4 Add 40mL of NaCl solution. Shake for 2min and discard the aqueous layer.
- 12.4.1.5 Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a Rotovap flask. Rinse the funnel with the sodium sulfate with two 15mL portions of hexane, add the rinses to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath).



### 12.4.2 Silica Column Cleanup

12.4.2.1 Preparation of Acid/Base silica gel columns for sample cleanup (as needed). Pack a Fresno Scientific customized glass tube with the following materials from bottom to top:

Glass wool plug  
Activated silica gel (1.0g)  
Basic silica gel (2.0 g)  
Activated silica gel (1.0g)  
Acidic silica gel (10g)  
Anhydrous sodium sulfate (1.0g)

12.4.2.2 Elute the column with 50mL hexane. Be careful not to let the column run dry.

12.4.2.3 Dissolve the sample residue in 2.0mL of hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4mL) to complete transfer of sample. Elute the silica gel column with 90mL of hexane.

12.4.2.4 Concentrate the eluent on a rotary evaporator (35°C water bath) to approximately 1mL.

### 12.4.3 Carbon Column Cleanup

12.4.3.1 Preparation of AX-21/Celite 545 carbon columns for sample cleanup (as needed). Pack a Fresno Scientific customized glass tube with the following materials from bottom to top:

Glass wool plug  
1cm plug of Celite 545  
1cm plug of the AX-21/Celite 545 Mixture  
1cm plug of Celite 545  
Glass wool plug

12.4.3.2 Rinse the AX-21/Celite 545 column with 5mL of toluene.

12.4.3.3 Rinse with 2.0mL of (75:20:5, v/v) Methylene chloride/methanol/toluene solution.

12.4.3.4 Rinse with 1mL of (1:1, v/v) Cyclohexane/methylene chloride solution.

12.4.3.5 Rinse with 5mL of hexane.

12.4.3.6 The flow rate should be less than 0.5mL/min. Discard the rinses.

12.4.3.7 While column is still wet with hexane, add the sample concentrate.

12.4.3.8 Rinse the concentrator tube twice with 1mL portions of hexane.

12.4.3.9 Rinse column sequentially with two 2mL portions of hexane, 2mL Cyclohexane/Methylene chloride (50:50, v/v), and 2mL-methylene chloride/methanol/toluene (75:20:5, v/v). Combine these eluents: This combined fraction may be used as a check on column efficiency.

12.4.3.10 Turn the column upside down and elute the PCDD/PCDF fraction with 20mL of toluene. Add the rinse to the eluent.



- 12.4.3.11 Concentrate the extract in a rotary evaporator (50°C water bath), to near dryness. Solvent transfer to 1.0mL of hexane.
- 12.4.4 Micro-Concentration: Quantitatively transfer the sample extract from the boiling flask to a 15mL centrifuge tube. Rinse out flask with two 5mL portions of hexane and add it to the centrifuge tube. Concentrate the extracts using a nitrogen blow-down apparatus.
- 12.4.4.1 Adjust the flow of the Nitrogen gas until the surface of the solvent is just visibly disturbed. When the volume of the eluent has concentrated to approximately 100µL, transfer the concentrate extract into a 300µL conical injection vial (marked at the 50µL level) for further concentration. Rinse centrifuge tube three times with 300µL of hexane. Between rinses, continue concentrating to 100µL volume and transfer concentrate to injection vial.
- 12.4.4.2 Add 20µL of 100ng/mL recovery standard and 30µL of nonane to the extract and continue concentrating under nitrogen until the 50µL level is reached. The sample is now ready for instrument analysis.
- 12.5 Recommended Operating Conditions for HRGC:  
Column: Carrier gas (He) flow rate: 1 mL/min  
Oven Temperature: 200 °C  
Equilibration Time: 1.0min  
Initial Temp: 200 °C  
Initial Time: 2min  
Temp Ramp: 5 °C/min to 220°C and hold 16 min, ramp 5°C/min to 235°C and hold 7 min, then ramp 5°C/min to 330°C and hold 5 min.  
Total Run Time: 60 min  
Injector Temp: 270 °C  
Interface Temp: 290 °C  
Injection Volume: 1µL
- 12.6 These conditions may be used as guidelines to establish an optimal GC temperature program. With the possible coelution of sample components, it may be necessary to adjust chromatographic conditions to give adequate separation of the characteristic peaks between individual congener peaks. Once a temperature program has been established, all samples must be analyzed under the same operating conditions as standards.
- 12.7 The HRGC run sequence should be arranged in the following manner:  
GC Column Performance Check Solution  
CS-0.2  
CS-1  
CS-2  
CS-3  
CS-4  
CS-5  
Nonane Blank  
GC Column Performance Check Solution  
CS-3 (CCV)  
12 hour analytical shift (including blanks, spikes and samples)  
CS-3 (CCV)



### 12.8 Instrument Calibration for Quantitative Analysis

12.8.1 One of the concentrations will be at the quantitation limit. The analyst must refer to the incoming sample notice for the lab works code and look at the detection limits listed on the appropriate form 1 to determine the quantitation limit standard. The initial calibration curve is a reflection of the performance of the instrument at any given time. Compounds react to the changing dynamic of the instrument. Therefore it is sometimes necessary to delete levels for compounds in an initial calibration curve. When this occurs the following rules are followed to ensure integrity of the data:

12.8.1.1 A standard must be included in the curve for each compound, which is less than or equal to the reporting limit. If the responses of a sample peak exceed the calibration range of the system, dilute the extract and reanalyze.

12.8.1.2 The deletion of discrete points must never result in a calibration curve consisting of less than five points for each analyte of interest.

12.8.1.3 Points for an individual analyte in the middle of the curve may not be deleted, however unforeseen circumstances may occur such as a miss injection by the autosampler, a loose cap on an injection vial, etc. In this situation the entire level is deleted for all compounds and the reason for deletion is noted on the multilevel form. If this results in a calibration curve that consists of less than five points, another level may be run before the analysis of samples begin.

12.8.1.4 Points at the low end and high end of the curve may be deleted if it is determined the compound ceases to be linear at either end. Any positive findings in the samples will be analyzed so as to fall within the linear range of that particular compound.

12.8.2 Tuning with PFK – The PFK ions monitored in the tune are 304.9825 m/z and 380.9760 m/z. Acceptance criteria is > 10,000 resolution.

12.8.3 Initial Calibration (ICAL) Analyze the five calibration standards (see Table 1) by HRGC. Record the sum of the peak areas for each of the two m/z of interest for each congener. The results can be used to prepare a calibration curve for each analyte. The ratio of the response to the amount injected, defined as the response factor (RF), can be calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average response factor can be used in place of a calibration curve. When this criterion is exceeded, inspect the HRGC system to determine the cause and perform whatever maintenance is necessary before re-calibrating and proceeding with analysis. The following criteria must also be met for the ICAL in order for sample analysis to proceed, otherwise the mass spec will need to be adjusted and the ICAL repeated.

12.8.3.1 The signal to noise ratio for each native and C<sub>13</sub> labeled standard must be ≥10.

12.8.3.2 The ion abundance ratios must be within acceptance method criteria (See Table 5).

12.8.3.3 The %RSD for native compounds is equal or less than 20%, and the %RSD for C<sub>13</sub> labeled compounds is equal or less than 30%.



12.8.3.4 Internal Standard Calibration Technique is used to determine PCDD/PCDF C<sub>13</sub> surrogates.

12.8.3.4.1 Calibration is achieved using the average response factors of the fixed concentrations of C<sub>13</sub> surrogates in each of the points of the calibration curve (See Table 1). The nearest-eluted C<sub>13</sub> I.S. is used for quantitation for each C<sub>13</sub> surrogate. The %RSD for the labeled standards should be 30% or lower.

12.8.3.4.2 Response factors are calculated as follows:

$$\text{Response Factor (RF)} = \frac{A_s C_{is}}{A_{is} C_s}$$

Where:

A<sub>s</sub> = Sum areas of both m/z for the C<sub>13</sub> surrogate

A<sub>is</sub> = Sum areas of both m/z for the IS

C<sub>s</sub> = Quantity of C<sub>13</sub> surrogate (pg)

C<sub>is</sub> = Quantity of IS (pg)

12.8.3.5 Isotope Dilution Technique is used to determine the native PCDD/PCDF compounds.

12.8.3.5.1 Calibration is achieved using the average response factors of the increasing concentrations of native PCDD/PCDF in the initial calibration curve. The corresponding C<sub>13</sub> surrogate is used to quantitate each native compound in the samples and spikes. The %RSD for the native standards average RF should be 20% or lower.

12.8.3.5.2 Response factors are calculated as follows:

$$\text{Relative Response Factor (RR)} = \frac{A_n C_L}{A_L C_n}$$

Where:

A<sub>n</sub> = Sum areas of both m/z for the native PCDD/PCDF

A<sub>L</sub> = Sum areas of both m/z for the C<sub>13</sub> PCDD/PCDF

C<sub>n</sub> = Quantity of native calibration standard (pg)

C<sub>L</sub> = Quantity of C<sub>13</sub> surrogate (pg)

The % RSD is calculated as follows:

$$\%RSD = (SD)(100\%)/(RF_{x1})$$

Where:

%RSD = Percent relative standard deviation.

RF<sub>x1</sub> = Mean of the initial RR for a compound.

SD = Standard deviation of the average RRF for a compound

12.9 Continuing Calibration Verifications (CCVs) must be analyzed at the beginning and end of each 12-hour analytical shift, by injecting the CS-3 calibration standard. The following CCV acceptance criteria must be met in order for analysis, otherwise the mass spec must be adjusted and a fresh CCV prepared and analyzed until the acceptance criteria



can be met. If the mass spec adjustment includes changing the resolution, then a new resolution check must also be prepared and analyzed. If these corrective actions are not successful, then a new ICAL must be prepared and analyzed.

12.9.1 The ion abundance ratios must be within acceptance method criteria (See Table 5).

12.9.2 For the beginning and ending CCV, the %D of the native PCDD/PCDF should be within 20% from the average RF from the ICAL. The %D of the C<sub>13</sub> labeled compounds should be within 30% from the average RF from the ICAL.

$$\%D = A - B / A$$

D = Difference

A = Average RF from the ICAL

B = RF from the CCV

NOTE: The method allows for marginal %D failures in certain cases, such as the C<sub>13</sub> labeled compound has a %D outside control limits while the corresponding native compound is acceptable.

12.10 Each sample analysis sequence must include an acceptable initial calibration; calibration verification standards and resolution check standards. When a CCV or Resolution Check fails to meet the acceptance criteria, all samples that were injected after the last acceptable check must be re-injected. For DoD QSM 5.0 clients: If the continuing calibration verification does not meet the requirements, recalibrate, and reanalyze all affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.

12.11 Sample injection may continue for as long as all the calibration verification standard requirements listed above are met.

### 12.12 Mass Spec Resolution

12.12.1 Using Perfluorokerosene (PFK) and a molecular leak, tune the instrument to meet resolution 10,000 (10% valley) at high reference m/z 380.9760 and low reference m/z 304.9824.

12.12.2 PFK is used to correct for mass-drift that may occur during the long analysis time. The lock-mass is established from PFK and is dependent on the m/z monitored within each descriptor. The deviation between the m/z values listed in the method and the actual m/z values must be less than 5ppm.

12.12.3 Resolution checks must be analyzed at the beginning and end of each analytical shift or at least every 12 hours. The MassLynx software includes a feature called "Experimental Calibration" that meets the resolution check method requirement. If the MS resolution criteria listed above can not be met, then analysis may not proceed. Samples affected by poor closing resolution check must be re-analyzed.

### 12.13 GC Performance Check

12.13.1 At the beginning of each 12 hour analytical shift a performance check standard will be run. See table 2. The peak to valley resolution for the tetra dioxin must be <25% of the highest peak in the chromatogram.



- 12.14 Qualitative Determination: A native or labeled congener is identified in a standard, blank or sample when all of the following criteria are met:
- 12.14.1 The signals for the two m/z isomers must be present and must maximize within  $\pm 2$  seconds of the two scans. The area ratio of the two ions must be within the criteria listed in Table 5 of this SOP for positive identification to be determined.
  - 12.14.2 The S/N ratio at the m/z of interest must be greater or equal to 2.5 for each compound detected in the sample extract and greater or equal to 10 for all congeners in the ICAL and CCV standards.
  - 12.14.3 Retention times are crucial to the identification of target congener compounds. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. The RT of a native compound must be within (-1 to +3) seconds of the corresponding C<sub>13</sub> compound. If the native compound does not have a corresponding C<sub>13</sub> compound (as shown in Table 1), then the RT of the native must fall within 0.005 units of the relative RT measured in the nearest CCV.
  - 12.14.4 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their retention time windows, the system is out of control. Determine the cause of the problem and correct it.
- 12.15 Homolog Totals by LOC Estimation: The total concentration of all PCDD/PCDFs at a given Level of Chlorination may be reported by summing the concentrations of all congeners detected at that LOC.
- 12.16 Toxicity Equivalent Concentration (TEQ): The TEQ for a particular environmental sample may be determined by summing the concentrations of each individual toxic PCDD/PCDF multiplied by their respective Toxicity Equivalent Factors (TEF) as described in the EPA method 8290<sup>1</sup>.
- 12.17 Manual integration: The TargetLynx software program is used for integration and quantitation of Dioxin / Furan data. Manual integration of peaks should be consistent with the ICAL integrations for each standard level. If method acceptance criteria are not met for a particular analyte, then examine the software's integration for that analyte, and determine whether or not the integration is consistent with the ICAL integration at each level. If not, then perform the appropriate manual integration. It may be necessary to integrate other levels also to keep the integration consistent. If it is not possible to meet the criterion while maintaining the same baseline, make any adjustments necessary and recalibrate the instrument. If method acceptance criteria are met, but the software's integration for a peak is inconsistent with the ICAL integration at each level and performing a manual integration would not cause the ICAL to be unacceptable, do not manually integrate. It is the intent of the laboratory to minimize the amount of manual integrations performed by the chemist. The TargetLynx software shows the "before" integration with a dashed line, and the "after" integration with a bold line on the chromatogram. Note a "M" followed by the number for the reason (see list below) a new integration was performed next to the peak on the chromatogram and initial and date. Save the manual integration electronically so that it can be retrieved at a later date if necessary. Place the chromatogram with the manual integration(s) in the data folder to be reviewed by the section manager or his/her designee.



- MI1) Integration does not follow baseline
- MI2) Non-target peak interference
- MI3) To split a peak that was integrated as one peak by the computer.
- MI4) To integrate a split peak
- MI5) The whole peak or part of the peak was not integrated.
- MI6) Computer integrated wrong peak
- MI7) Other – (See case narrative)

After review by the section manager, the manager will date and initial. Upon client request, the integrations will be reviewed by the QAU or his/her designee initialed and dated. The hard copies will be filed with the raw data.

12.18 For any DoD samples, please refer to Table 8 for client specific criteria.

### 13.0 Data Analysis and Calculations

13.1 The sample and spikes are calculated against the initial calibration curve for the native PCDD/PCDF using isotope dilution. The C<sub>13</sub> labeled surrogates are calculated using the two recovery standards, C<sub>13</sub>-1,2,3,4-TCDD and C<sub>13</sub>-1,2,3,7,8,9-HxCDD.

13.2 Quantitation algorithm check: The quantitation of the analytes is performed by the Waters Data System. The algorithm is checked at least once per computer file (daily) by calculating the amount of analyte injected from the peak response, using the calibration curve. The following calculation is used to check the quantitation:

$$13.2.1 \text{ Concentration (pg/L or pg/g) Isotope Dilution Technique} = \frac{(A_s)(C_L)}{(A_i)(RF)(V_o)}$$

Where:

A<sub>s</sub> = Sum areas of both m/z of analyte

A<sub>i</sub> = Sum areas of both m/z of internal standard

C<sub>L</sub> = Labeled Congener quantity (pg)

RF = Relative Response Factor of analyte from ICAL (see Sect 7.5.6)

V<sub>o</sub> = Volume/weight of sample extracted (L or g)

13.3 Reporting Positive Findings: The Target Lynx software calculates the LOD and EMPC values, which are dynamic and dependent on the S/N ratio of each sample. The laboratory establishes the PQL, and it is a static value based on the lowest point in the calibration curve. J-values are reported between the LOC or EMPC and the PQL according to the following criteria:

- 13.3.1 If there is no peak present in the sample, then report “not detected” along with an LOD (or EDL) value in the EMPC/EDL column in Labworks.
- 13.3.2 If there is a peak present in the sample, but it does not meet all the criteria for RRT or Isotope Ratio, then report “not detected” along with the EMPC value.
- 13.3.3 If there is a peak in the sample that is a confirmed hit above the PQL, then report that value along with the EMPC value listed in the EMPC/EDL column in Labworks.



13.3.4 If a peak exists but have a signal to noise ratio below 2.5, then report “not detected” along with the EDL value.

13.3.5 LOD (Limit of Quantitation) or EDL (Estimated Detection Limit)

$$13.4 \text{ LOD/EDL} = \frac{2.5 \times (H_x^1 + H_x^2) \times Q_{is}}{(H_{is}^1 + H_{is}^2) \times W \times \text{RRF}}$$

Where:

$H_x^1$  and  $H_x^2$  = peak heights of the noise level for both quantitation ions of the native

$H_{is}^1$  and  $H_{is}^2$  = peak heights of the noise level both quantitation ions of the appropriate IS

$Q_{is}$  = quantity of the appropriate IS injected (pg)

$\text{RRF}_n$  = calculated RRF from the CCV

$W$  = volume or weight of sample extracted in liters or grams.

13.5 EMPC (Estimated Maximum Possible Concentration)

$$\frac{Q_{is} \times (A_x^1 + A_x^2) \times D}{V \times (A_{is}^1 + A_{is}^2) \times \text{RRF}}$$

Where:

$A_x^1$  and  $A_x^2$  = areas of both quantitation ions of the noise. For the EMPC, this represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

$A_{is}^1$  and  $A_{is}^2$  = integrated areas of both quantitation ions of the appropriate IS

$D$  = Dilution Factor

$Q_{is}$  = quantity of the appropriate IS injected (pg)

$\text{RRF}_n$  = calculated RRF from the CCV

$V$  = volume or weight of sample extracted

13.6 PQL (Practical Quantitation Limit)

$$\text{PQL} = \text{CS}_1 \times E \times D$$

Where:

$\text{CS}_1$  = concentration of low calibration standard (pg/mL)

$E$  = extraction ratio (for waters: 0.05mL / 1L) and (for soils (0.05mL / 10g)

$D$  = Dilution Factor

13.7 Using the MassLynx Program (software v4.1 SCN627)

13.7.1 Quantitation methods are built in the MassLynx program, according to specific criteria stated in each EPA method (8290 or 1668).

13.7.2 The quantitation method is then used to calculate an initial calibration. The resulting responses for the initial calibration are then used to calculate results for samples and QC.



13.7.3 Retention Time and Signal to Noise Forms are generated for data interpretation purposes.

13.7.4 There are two different formats by which data can be quanted: QuanLynx and TargetLynx. For this analysis, be sure to highlight TargetLynx. It is located at the left-hand side of the MassLynx window.

File Name	File Text	Type	A	B	C	D	E	F	G	H	I	DF
091129_HR_01	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_04	EDF-9999 CS-1 04/28/09	Standard	0.5	2.5	2.5	5	100	100	200	100	100	1.00
091129_HR_05	EDF-9999 CS-2 04/28/09	Standard	2	10	10	20	100	100	200	100	100	1.00
091129_HR_06	EDF-9999 CS-3 11/16/09	Standard	10	50	50	100	100	100	200	100	100	1.00
091129_HR_07	EDF-9999 CS-4 04/28/09	Standard	40	200	200	400	100	100	200	100	100	1.00
091129_HR_08	EDF-9999 CS-5 04/28/09	Standard	200	1000	1000	2000	100	100	200	100	100	1.00
091129_HR_11	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_12	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_14	091123SA_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_15	091123SA_LCSD-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_17	091123SA_BLK 5.000 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_18	AY07842_S01 0.820 DF 11/23/09	Analyte					40	100	200	40	40	20.00
091129_HR_19	AY07843_S01 0.760 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_20	AY07844_S01 2.270 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_21	AY07845_S01 0.870 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_22	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_23	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_26	AY07846_S01 0.660 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_27	091123SA_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_28	091123SA_LCSD-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_30	091123SA_BLK 5.000 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_31	AY07842_S01 0.820 DF 11/23/09	Analyte					40	100	200	40	40	20.00
091129_HR_32	AY07843_S01 0.760 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_33	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_34	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_36	AY07844_S01 2.270 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_37	AY07845_S01 0.870 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_38	AY07846_S01 0.660 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_40	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_41	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00

### 13.7.5 Building a Method

13.7.5.1 In the TargetLynx format, click on the 'Edit Method' icon. The method editor window should pop up. The most recent method that was previously viewed should be displayed.

13.7.5.2 It is best to do a 'Save As' to make sure any previous methods are not overwritten. Under 'File' → 'Save As' → method name.

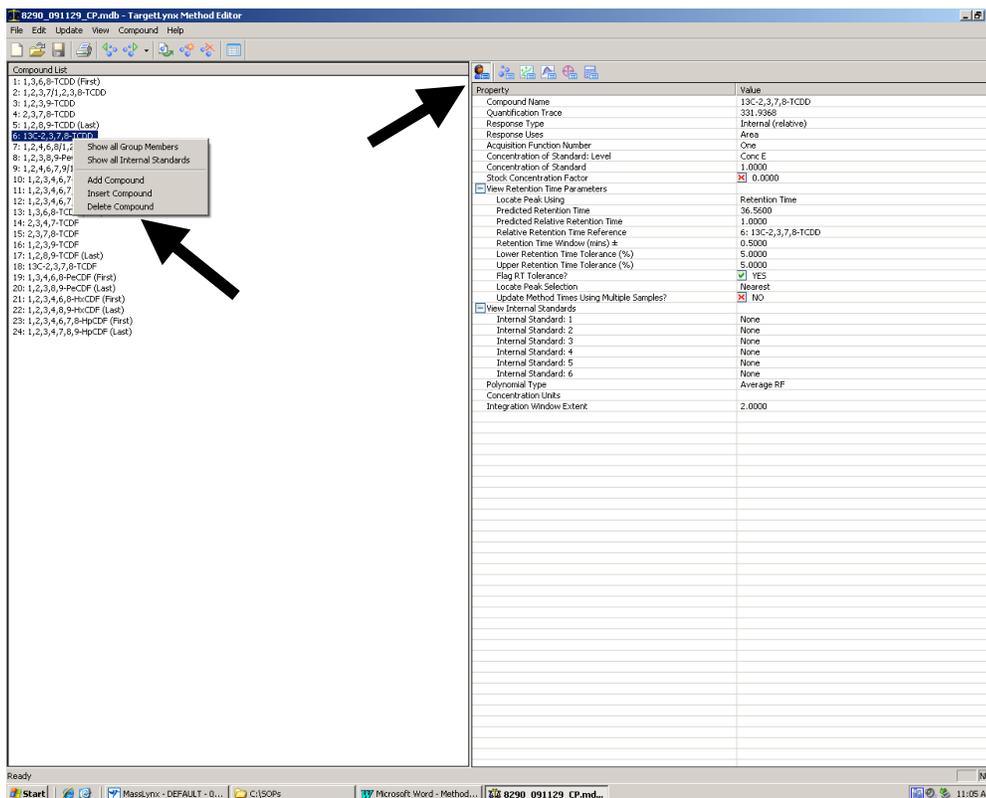
13.7.5.3 The format for which the method should be saved is in the following order: the method name, date and method type. For example, an 8290 method on Nov 1<sup>st</sup>, 2009 would be: 8290\_091101. In the case of an 8290 column performance method on the same date, the proper name would be" 8290\_091101\_CP.

13.7.5.4 The TargetLynx Method Editor has an extremely large number of parameters with which to build the method. It is nearly impossible to



list and explain every single parameter. The goal of this SOP is to target the main parameters that are used in the 8290 methods.

13.7.5.5 To the left of the Method Editor window is the compound list.



Right clicking and selecting 'Show all Group Members' will highlight the compounds that are linked with the selected compound. Selecting 'Show all Internal Standards' would highlight the specific internal standard that the compound is linked to. Designating groups and internal standards will be explained later in the SOP. Selecting the 'Add Compound' would add a new compound at the end of the list. The 'Insert Compound' option would add a new compound just above any highlighted compound.

13.7.5.6 To the right of Method Editor are the six different property parameters of the compound: User-defined Properties, Compound Properties, Calibration Properties, Integration Properties, Targeting Properties, and Calculation Factors.

### 13.7.5.7 User-defined Properties

13.7.5.7.1 Enter the 'Compound Name' and 'Quantification Trace' for the specific compound.

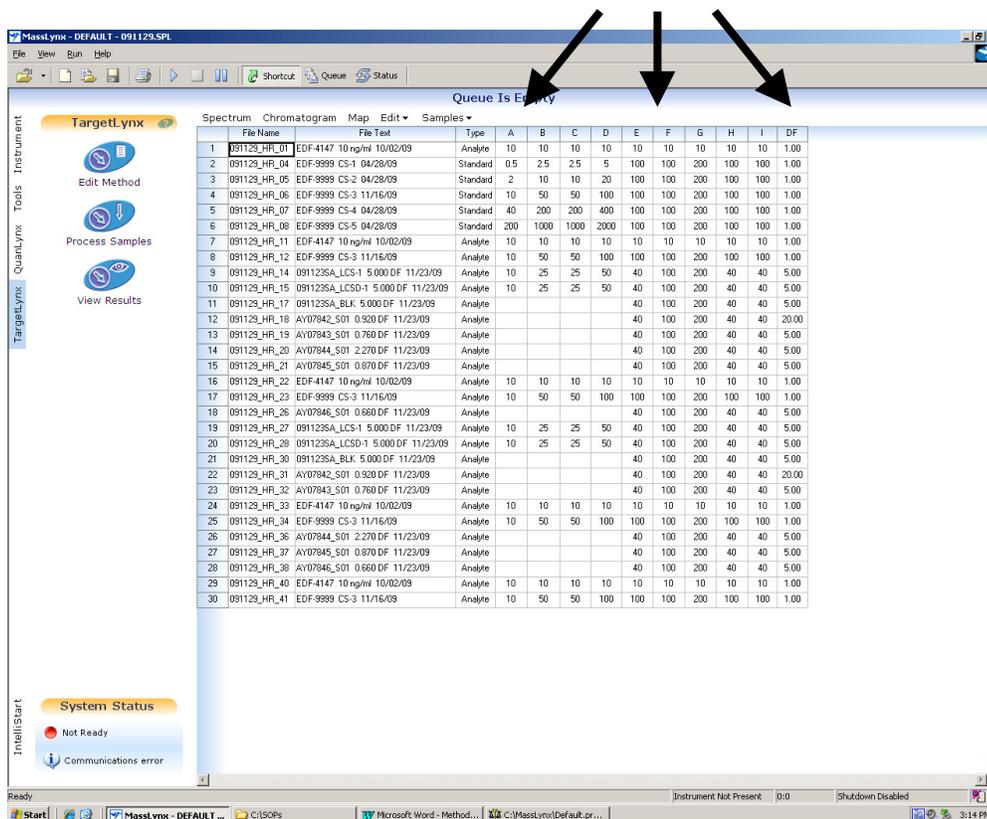
13.7.5.7.2 The 'Response Type' should be set to 'Internal (relative)' and the 'Responses Uses' should be set to 'Area'.

13.7.5.7.3 The 'Acquisition Function Number' is dependent on the actual function method of the HR-MS. Usually in 8290 analysis, any tetra compounds would be in the first function, the penta compounds in the second function, and so forth. For method



1668, this is not the case. For example, a tetra compound might be found in the sixth function.

13.7.5.7.4 The 'Concentration of Standard: Level' will use whatever value is set in the sequence list. Usually in an 8290 method, columns A through D are the spike concentrations of tetra through octa groups. Columns E through G are surrogate concentrations while columns H and I are internal standard concentrations.



13.7.5.7.5 The 'Concentration of Standard' is set to '1.000' and the 'Stock Concentration Factor' is 'X' out.

13.7.5.7.6 For target analytes and surrogates in method 8290, the 'Locate Peak Using' option is set to 'Relative Retention Time.' The only compounds that will use 'Retention Time' would be the internal standards.

13.7.5.7.7 The 'Predicted Retention Time' is not used for the target and surrogate analytes. However, it is required for the internal standards.

13.7.5.7.8 The 'Predicted Relative Retention Time' is determined by taking the ratio of the retention time of the analyte to its surrogate/IS. Use the midpoint of the initial calibration to determine the ratio.

13.7.5.7.9 The 'Relative Retention Time Reference' are the surrogate/IS compounds to the target analytes.



- 13.7.5.7.10 'Retention Time Windows' are decided by the user. Keep in mind that a narrow window might not allow for the target analyte to be picked up while a broad window will pick up too many peaks. Values that seem to do very good for 8290 and 1668 analysis are  $\pm 0.200$  min for the retention time windows and lower/upper retention time tolerance of 0.500%. The retention time checks will be done later using Excel.
- 13.7.5.7.11 The 'Flag RT Tolerance' option is usually checked.
- 13.7.5.7.12 The 'Locate Peak Selection' is always set to 'Nearest'.
- 13.7.5.7.13 The 'Update Method Times Using Multiple Samples' is 'X' out.
- 13.7.5.7.14 In the internal standard:1 option, enter the corresponding surrogate/IS for the compound. In method 8290, there is only one surrogate/IS for each analyte. In method 1668, a compound could have multiple internal standards.
- 13.7.5.7.15 The 'Polynomial Type' is set to 'Average RF'.
- 13.7.5.7.16 The 'Integration Window Extent' is set to '2.0000'.
- 13.7.5.8 Compound Properties – there will be parameters in this section that would already be inputted from the User-defined Properties section.
- 13.7.5.8.1 The 'Include Quan Trace in Response' and 'Use absolute mass window' options should be checked 'Yes'.
- 13.7.5.8.2 The 'View Acceptance Flag Parameters' section should all been 'X' out and labeled 'No'.
- 13.7.5.8.3 Each compound can be designated a totals group. In method 8290 names of totals include: TD, PD, HxD, HpD, TF, PF, HxF, and HpF. The 'Symmetry Threshold (%)' is usually set to '90.00'.
- 13.7.5.9 Calibration Properties - there will be parameters in this section that would already be inputted from the User-defined Properties section.
- 13.7.5.9.1 The 'Calibration Origin' should be set to 'Exclude'.
- 13.7.5.9.2 Both 'Weighting Function' and 'Axis Transformation' parameters should be set to 'None'.
- 13.7.5.9.3 The 'User RF Value', 'Minimum Coefficient of Determination', and 'Maximum Relative Response Standard Deviation' should all be 'X' out.
- 13.7.5.9.4 The 'Propagate Calibration Parameters' should be check 'Yes'.
- 13.7.5.10 Integration Properties
- 13.7.5.10.1 The 'Smoothing Enabled' should be checked 'Yes'.
- 13.7.5.10.2 The 'Smoothing Method' should be set to 'Mean.'
- 13.7.5.10.3 The 'Smoothing Iterations' and 'Smoothing Width' parameters are dependent on the quality of the chromatography. To reduced the number of manual Q-edits, the values are usually both set to '4' and '2'.
- 13.7.5.10.4 The "Apex Track Enabled?" is checked 'Yes'.
- 13.7.5.10.5 The 'Peak-to-Peak Baseline Noise' and 'Peak With at 5% Height' options are checked at a value of '5'.
- 13.7.5.10.6 The 'Baseline Start Threshold %' and 'Baseline End Threshold' options are set to '0'.
- 13.7.5.10.7 The 'Detect Should Peaks' option should be checked 'Yes'.



- 13.7.5.10.8 The 'Automatic Noise Measurement' option should be 'X' out and labeled 'No'.
- 13.7.5.10.9 The 'Minimum Signal/Noise Ratio' is usually set to '2.5'.
- 13.7.5.10.10 The 'Flag Signal/Noise Ratio' option should be checked 'Yes'.
- 13.7.5.11 Targeting Properties
  - 13.7.5.11.1 The 'Calculate Ion Ratio Tolerance As' option should be set to 'Ratio'.
  - 13.7.5.11.2 The 'Target Ion Ratio Method' option should be set to 'Quan/Target'.
  - 13.7.5.11.3 The 'Quantification Ratio' should be set to '1.000'.
  - 13.7.5.11.4 The 'Use trace in response calculation' should be checked 'Yes'.
  - 13.7.5.11.5 The 'Target Ion Ratio' and the 'Target Ion Ratio Tolerance' options are method specific.
  - 13.7.5.11.6 The 'Target Ion Must Exist' option is checked 'Yes' while the 'Target Ion Must Past Ratio' and 'Calculate Ion Ratio As (Target/Primary)' options are 'X' out and labeled 'No'.
- 13.7.5.12 Calculation Factors
  - 13.7.5.12.1 The 'Toxic Equivalence Factors' could be used to calculate the TEQ for each compound. However, keep in mind that each client may have their own specific TEF values.
  - 13.7.5.12.2 The 'Signal-to-noise method' should be set to 'Peak-to-Peak' and the 'Noise calculation factor' set to '1.000'.
  - 13.7.5.12.3 The 'Noise window start and end' should both be set to '0'.
  - 13.7.5.12.4 The 'Measure peak signal level from' should be set to 'Avg Noise Level'.
  - 13.7.5.12.5 The 'Detection Limit Factor' is client dependent. For method 8290, designate a signal to noise ratio of 2.5.
  - 13.7.5.12.6 The 'Quantitation Limit Factor' should to set to "1.000".
  - 13.7.5.12.7 Both the 'Propagate Detection Limit Settings' and 'Use EMPC' options should be checked 'Yes'.
- 13.7.6 Sequence Setup
  - 13.7.6.1 Different sections of the sequence can be processed by highlighting the data files and clicking 'Process Samples'. It is not possible to highlight different sections of a sequence at the same time. The only way to do this is to quant different sections one at a time. This will create numerous datasets.



# Standard Operating Procedure

## QA Control Copy #   3

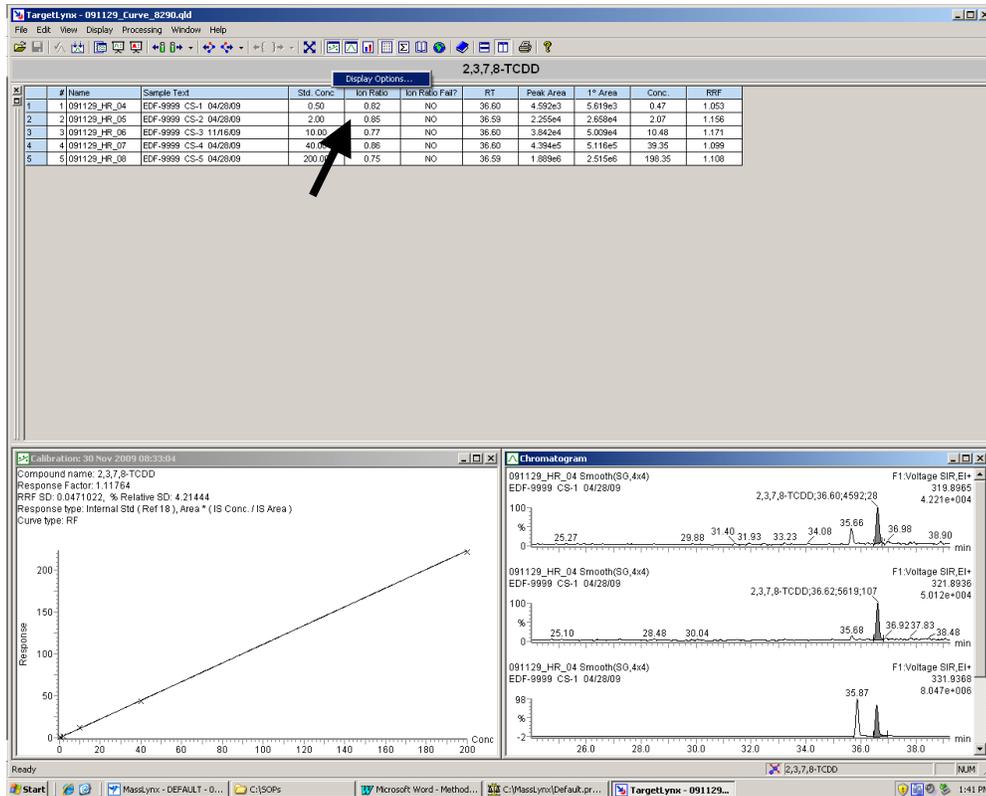
SOP: HPL8290  
Section: 10  
Revision: 11  
Date: 07/23/14

File Name	File Text	Type	A	B	C	D	E	F	G	H	I	DF
091129_HR_01	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_04	EDF-9999 CS-1 04/28/09	Standard	0.5	2.5	2.5	5	100	100	200	100	100	1.00
091129_HR_05	EDF-9999 CS-2 04/28/09	Standard	2	10	10	20	100	100	200	100	100	1.00
091129_HR_06	EDF-9999 CS-3 11/16/09	Standard	10	50	50	100	100	100	200	100	100	1.00
091129_HR_07	EDF-9999 CS-4 04/28/09	Standard	40	200	200	400	100	100	200	100	100	1.00
091129_HR_08	EDF-9999 CS-5 04/28/09	Standard	200	1000	1000	2000	100	100	200	100	100	1.00
091129_HR_11	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_12	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_14	091129A_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_15	091129A_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_17	091129A_BLK 5.000 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_18	Ay07842_S01 0.920 DF 11/23/09	Analyte					40	100	200	40	40	20.00
091129_HR_19	Ay07843_S01 0.760 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_20	Ay07844_S01 2.270 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_21	Ay07845_S01 0.670 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_22	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_23	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_26	Ay07846_S01 0.660 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_27	091129A_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_28	091129A_LCS-1 5.000 DF 11/23/09	Analyte	10	25	25	50	40	100	200	40	40	5.00
091129_HR_30	091129A_BLK 5.000 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_31	Ay07842_S01 0.920 DF 11/23/09	Analyte					40	100	200	40	40	20.00
091129_HR_32	Ay07843_S01 0.760 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_33	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_34	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00
091129_HR_36	Ay07844_S01 2.270 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_37	Ay07845_S01 0.670 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_38	Ay07846_S01 0.660 DF 11/23/09	Analyte					40	100	200	40	40	5.00
091129_HR_40	EDF-4147 10 ng/ml 10/02/09	Analyte	10	10	10	10	10	10	10	10	10	1.00
091129_HR_41	EDF-9999 CS-3 11/16/09	Analyte	10	50	50	100	100	100	200	100	100	1.00

From the above sequence, data 091129\_HR\_12 thru 091129\_HR\_21 will be quanted to generate a new dataset.

### 13.7.7 Processing the Method

- 13.7.7.1 Highlight the data files that will be processed.
- 13.7.7.2 Click on the 'Process Samples' icon on the left side of the screen.
- 13.7.7.3 Under the 'Operations' options, the 'Integrate Samples', 'Calibrate Standards', and 'Quantify Samples' parameters should all be check marked.
- 13.7.7.4 Under the 'Quantify' options, the method that should be loaded is the one saved for the sequence. The curve parameter would be highlighted.
- 13.7.7.5 Click on 'OK'. A data set would be generated and a 'TargetLynx – untitled' window will pop up.
- 13.7.7.6 In the 'TargetLynx – untitled' window, click on 'File' → 'Save As'. For curve data sets, the data on date 11-29-09 is save as 091129\_Curve\_8290.
- 13.7.7.7 There are different layouts by which a data set could be displayed. To apply the curve format, click on File → Apply Layout → pick Layout\_Curve\_8290.
- 13.7.7.8 Right clicking on the name header will give some display options. Go to the summary tab and select 'List by compound'. This will display each compound with calibration and chromatograph data.



13.7.7.9 The green injection vial icons are used to scroll through each data file. The blue and red dot icons are used to scroll through the analytes. For both of these icons, use the left / right arrows to scroll back and forth through injections or analytes.

13.7.7.10 Two of the main parameters that need to be reviewed are the ion ratios and the %RSD. The ion ratios are located on the display options window. The ion ratio and whether or not it passes method criteria is displayed in the table. This is shown under the column 'Ion Ratio Fail?' 'No' means the ion ratio passes, and 'Yes' means the ion ratio fails. The %RSD is shown in the calibration window. Methods 8290 and 1668 require a %RSD of 20% or lower. Check the peak integrations in order to make sure the software integrated the peaks properly, according to APPL Inc. protocol.

13.7.7.11 Once a passing initial calibration is achieved, click on 'File' → 'Save As' → 091129\_Curve\_8290. The name of the curve is date dependent.

13.7.7.12 The calibration data also needs to be exported to a file for future use. Click on 'File' → 'Export' → 'Calibration' → '091129\_8290'.

### 13.7.8 Processing the Samples

13.7.8.1 Highlight the samples that are to be processed, and click on the 'Process Samples' icon on the left side of the screen.



- 13.7.8.2 Under the 'Operations' options, only the 'Integrate Samples' and 'Quantify Samples' parameters should be checked marked.
- 13.7.8.3 Under the 'Quantify' options, load the saved method pertinent to the current sequence. The curve parameter is the data file that was saved during the exportation of the calibration curve.
- 13.7.8.4 Click on 'OK'. A data set will be generated and a 'TargetLynx – untitled' window will pop up.
- 13.7.8.5 In the 'TargetLynx – untitled' window, click on 'File' → 'Save As' and save the data set using the following convention: for example, the data from date 11-29-09 would be saved as 091129\_Samples\_5-10\_8290. The '5-10' represents the data files being quanted.
- 13.7.8.6 There are different layouts by which a data set could be displayed. To apply the sample format, click on 'File' → 'Apply Layout' → pick 'Layout\_Samples\_8290'. To apply the signal to noise format, click on 'File' → 'Apply Layout' → pick 'Layout\_SignalNoise'. To apply the retention time format, click on 'File' → 'Apply Layout' → pick 'Layout\_RRT'.
- 13.7.9 Processing for Column Performance
- 13.7.9.1 Quantify the column performance standard (EDF-4147) using method 8290\_091129\_CP. The '091129' represents the date in which the column performance method was saved.
- 13.7.9.2 Highlight the samples that are to be processed and click on the 'Process Samples' icon on the left side of the screen.
- 13.7.9.3 Under the 'Operations' options, the 'Integrate Samples', 'Calibrate Standards', and 'Quantify Samples' parameters should all be check marked.
- 13.7.9.4 To apply the column performance format, click on 'File' → 'Apply Layout' → pick 'Layout\_CP'.
- 13.7.9.5 Print the column performance data.
- 13.7.9.6 To display the chromatogram, highlight the sample in the MassLynx sequence. Click on 'Chromatogram' above the sequence table and another window will appear. Click on 'Display' → 'Mass' then pick the appropriate function and mass. For tetra-dioxins, use the 319.1985 m/z ion. For tetra-furans, use the 303.9016 m/z ion. Print the chromatogram.
- 13.7.10 Retention Time Form Generation
- 13.7.10.1 Load the dataset and apply the retention time format, click on 'File' → 'Apply Layout' → pick 'Layout\_RRT'.
- 13.7.10.2 To generate the data to notepad, click on 'File' → 'Export' → 'Complete Summary'. Save to H:\Magneto\Raw data for RRT. Paid attention to the format of the data being saved.
- 13.7.10.3 Load the notepad data, press Control A to select all, and then paste into an excel spreadsheet.
- 13.7.10.4 Load a previous retention time check under H:\MAGNETO\8290 Forms\Method 8290B\RRT\_Form\_....
- 13.7.10.5 Locate the continuing in the raw data and copy and paste the RRT of the continuing under the column with the heading 'RRT of congener in CCV'. Make sure to just paste the values under the



- paste special format. Change the data file name under the header to reflect the correct data file.
- 13.7.10.6 Locate the sample in the raw data and copy and paste the 'RT', 'IS RT, and 'RRT' to the headers 'RT of congener in sample', 'RT of 13C congener in sample', and 'RRT of congener in sample'. Make sure to just paste the values under the paste special format. Change the data file name under the header 'RT of congener in sample'. The other headers will automatically reflect the change in data file.
  - 13.7.10.7 Type in the sample name.
  - 13.7.10.8 The 'Qualifiers' column will show if the analyte passes the required retention time windows.
  - 13.7.11 Signal to Noise Form Generation
    - 13.7.11.1 Load the dataset and apply the signal to noise format, click on 'File' → 'Apply Layout' → pick 'Layout\_SignalNoise'
  - 13.7.12 Data Interpretation
    - 13.7.12.1 Forms needed during data interpretation
      - 13.7.12.1.1 The sample format pages which includes the sample summary sheet and the ion chromatograms obtained from TargetLynx under 'Layout\_Samples\_8290'.
      - 13.7.12.1.2 The retention times pages which includes the excel sheet and the raw data obtained from TargetLynx under 'Layout\_RRT'.
      - 13.7.12.1.3 The signal to noise pages obtained from TargetLynx under 'Layout\_SignalNoise'.
    - 13.7.12.2 Q-edits - It may be necessary to scan through the ion chromatograms of each sample to determine if any q-edits are needed.
    - 13.7.12.3 Identification Criteria
      - 13.7.12.3.1 Retention Times
        - a. For any congener that have an Isotopically-labeled internal standard, the retention time of both primary and secondary ions must be within -1 to +3 sec of the Isotopically-labeled standard.
        - b. For any congener that do not have an Isotopically-labeled internal standard, the retention time must fall within 0.005 retention time units of the relative retention times of the continuing calibration verification.
        - c. The peaks for both primary and secondary ions of both the congener and surrogates must be +/- 2 sec.
        - d. Using the excel sheet that was generated from the relative retention times, look for any failures. Note any failures with a 'RT' on the sample summary sheet.
      - 13.7.12.3.2 Ion Abundance Ratios
        - a. The ion abundance ratios must not exceed the control limits set in Table 8.
        - b. The ion ratio pass/fail column is displayed in the sample summary sheet.
      - 13.7.12.3.3 Signal-to-Noise Ratio



- a. The signal to noise ratio for congeners must be  $S/N \geq 2.5$  times noise level.
  - b. The signal to noise ratio for labeled congeners must be  $S/N \geq 10$  times noise level.
  - c. The signal to noise pass/fail column is displayed in the sample summary sheet.
- 13.7.12.3.4 Polychlorinated Diphenyl Ether Interferences
- a. In order for a peak to constitute being a hit, there must not be any peaks in the polychlorinated diphenyl ether channel that's has  $S/N \geq 2.5$  times noise level and  $\pm 2$  seconds.
  - b. Contamination could be determined by looking at the ion chromatogram of the sample and comparing it to di-phenol ether channel.
- 13.7.12.3.5 Using EDL or EMPC - If the signal to noise ratio for a hit is below 2.5 or there is any retention time failure, report the EDL value. Otherwise, report the EMPC value.
- 13.7.13 Reporting the Data
- 13.7.13.1 It is not an option to direct upload data from the TargetLynx software directly in the LIMS system. An excel spreadsheet is set up so that raw data can be properly adjusted and then exported into LIMS.
- 13.7.13.2 Exporting raw data into excel
- 13.7.13.3 Load up the data set and apply the sample format, click on 'File' → 'Apply Layout' → pick 'Layout\_Samples\_8290'. This data should be in notepad format. Press 'Control A' then 'Control C' to highlight and copy everything.
- 13.7.13.4 Go to H:\MAGNETO\Labworks Ready Data\ and click on the last dated excel spreadsheet.
- 13.7.13.5 Click on the 'A1' cell and press 'Control V' to paste the data from notepad.
- 13.7.13.6 Save the data under the most recent date.
- 13.7.13.7 Column W will display the Labworks format of which the specific data is displayed. For example it might display \$8290S Sample or \$8290S Blk. In order to apply the right format for each sample, copy the data from columns R thru AC of one sample and paste it into another. The format of the cells should transfer over.
- 13.7.13.8 With the raw data in front of you, delete all non-hits from column K.
- 13.7.13.9 Delete all EDL values are used in column M. The EMPC values in column N do not need to be deleted because the spreadsheet will adjust accordingly for LOD/EMPC usage from column M.
- 13.7.13.10 Significant figures will need to be adjusted in columns Z and AA.
- 13.7.13.11 Change any values accordingly for the Dilution Factor, Sequence, Run, etc in column Z.
- 13.7.13.12 To add the DL or PC after the EDL/EMPC numbers, click on 'Tools' → 'Macro' → 'Macros' → 'ColumnAAtoAC\_Rounding



Macro'. This should generate the EDL/EMPC values with the DL/PC on column AC. Specific clients will sometimes request this format.

13.8 For any DoD samples, please refer to Table 8 for client specific criteria.

### 14.0 Data Assessment and Acceptance Criteria for QC

- 14.1 When QC parameters are exceeded, the following will take place: When the matrix spikes are outside of the limits they are re-digested and re-analyzed. When the LCS is outside of limits the entire batch is re-digested and re-analyzed. Due to the cost of ICAL and spike mix standards, the spike mix may be injected and the quantitated concentrations may be used to calculate spike recoveries for LCS and MS/MSD. If there is not enough sample for re-digestion the Project Manager is notified whom in turn notifies the client by phone or fax. The case narrative or case letter explains the sequence of events and the data is qualified. If the calibration parameters are not met the standards are re-prepared and reanalyzed.
- 14.2 The analyst completing the work first reviews data. The initial calibration curve is reviewed, the continuing calibration %D is reviewed, the spike recovery and precision is reviewed, the performance check solution is reviewed, and the closing continuing calibration %D is reviewed. If at any point the review shows an out of control situation, the section manager is notified verbally and the problem is investigated. The correction may be one of several points considered: standard preparation, improper injection size, extraction technique, etc. The problem is potentially solved and reanalysis or re-extraction/reanalysis is completed.
- 14.3 The second level of review is either by a peer in the same section or the section manager. There is a Multilevel Quality Control Sign Off worksheet that is filled out in its entirety by the review person.
- 14.4 When QC parameters are exceeded, the following will take place: When the matrix spikes are outside of the limits they are re-injected and the client is notified. When the LCS is outside of limits the entire batch is re-extracted and reanalyzed. If there is not enough sample for re-extract, the Project Manger is notified who in turn notifies the client by phone, fax, or e-mail. The case narrative or case letter explains the sequence of events and the data is qualified. If the calibration parameters are not met, the standards are re-prepared and reanalyzed.
- 14.5 TargetLynx is the program used to quant all generated data. Consequently, flagging of data is mostly done using the TargetLynx program. This is flagged either with a "No" or "Yes". In TargetLynx, the question asked when checking tolerance such is the target ratio is "Does the measured value fall outside the specified tolerance?" or in essence, "Does the test fail?" Thus the value outside of the tolerance is flagged with a "Yes." Those within tolerance are flagged with a "No." The program does not allow changing of the flags from YES/NO to FAIL/PASS. This information is noted to avoid any confusion involved in flagging of data.
- 14.6 Quick check List for acceptance of 8290 data:
- 14.6.1 Tuning
- 14.6.1.1 The instrument needs to main static resolving power  $\geq 10,000$  (10% valley) for identified masses per period.
- 14.6.1.2 This should be check at the beginning and the end of each 12-hour period of analysis.
- 14.6.2 GC column performance check



- 14.6.2.1 Needs to be performed prior to ICAL or CCV performance check solution
- 14.6.2.2 There must be at least 25% valley peak separation between 2,3,7,8-TCDD and other TCDD isomers.
- 14.6.2.3 All first and last eluters of the homologue retention time windows needs to be identified with a “F” or “L”
- 14.6.2.4 The absolute retention times for switching from one homologous series to the next is  $\geq 10$  seconds for all components of the mixture.
- 14.6.3 Initial calibration
  - 14.6.3.1 Ion abundance ratios need to pass according to Method 8290
  - 14.6.3.2 Signal to noise ratio is  $\geq 10$  for all target analytes.
  - 14.6.3.3 % RSD is  $\leq 20\%$  for all natives and labeled internal standards.
- 14.6.4 Calibration verification
  - 14.6.4.1 The calibration verification should be run at the beginning of each 12-hour period and at the end of each analytical sequence.
  - 14.6.4.2 Ion abundance ratios need to pass according to Method 8290.
  - 14.6.4.3 Signal to noise ratio is  $\geq 10$  for all target analytes.
  - 14.6.4.4 % RSD is  $\leq 20\%$  for all natives and  $\leq 30\%$  for labeled internal standards.
- 14.6.5 Internal standard
  - 14.6.5.1 The control limits for all internal standards is 40-135%.
- 14.6.6 Sample PCDD/PCDF identification
  - 14.6.6.1 For 2,3,7,8-substituted isomers with labeled standards, the absolute retention time should be within  $-1$  to  $+3$  seconds of the corresponding labeled standard.
  - 14.6.6.2 For 2,3,7,8-substituted isomers without labeled standards, the relative retention time should be within 0.005 RRT units of the retention time in the calibration verification standard.
  - 14.6.6.3 Primary and secondary ions should elute with  $\pm 2$  seconds of each other.
  - 14.6.6.4 Ion abundance ratios need to pass according to Method 8290.
  - 14.6.6.5 Signal to noise ratio is  $\geq 2.5$  for all target analytes and  $\geq 10$  for all labeled standards.
  - 14.6.6.6 There should be no signal present having a S/N ratio  $\geq 2.5$  or the corresponding ether (PCDPE) detected at the same retention time ( $\pm 2$  sec).
- 14.7 For any DoD samples, please refer to Table 8 for client specific criteria.

**15.0 Corrective Actions and Contingencies for Out of Control Data or Unacceptable Data**

In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a NWR. The QAU has the final authority to approve the actions taken



### 16.0 Method Deviations

This SOP was compared to EPA method 8290<sup>1</sup>. If the TCDF isomers listed in the method are resolved by 25% on the DB-5 column, then the confirmation column analysis is not necessary for 2,3,7,8-TCDF. The column performance mix purchased by APPL Inc. contains 2,3,4,7-TCDF and 1,2,3,9-TCDF in order to show the separation needed to eliminate the confirmation column analysis.

The method requirement will be followed for re-calibrating the instrument whenever a new CS-3 lot number is purchased, if the new lot number does not meet the 20%D CCV requirement. If the new lot number for CS-3 does meet the 20%D requirement, then the instrument will not be re-calibrated, due to the high cost of 8290 prepared standards.

### 17.0 Pollution Prevention

All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes-acidic, basic and solvents.

### 18.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and lands disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

### 19.0 Method Performance

19.1 Continuing method performance is monitored by analysis of LCS samples with each batch and control charting the results as per SOP# QC016.

19.2 A method detection Limit (MDL) study is run to ensure the performance of the instrumentation is able to satisfy data quality objectives of the client by reaching the reporting limits necessary. An MDL study is performed for each matrix per instrument after major instrument changes take place, such as a column change and is performed in accordance with SOP# QC018.

19.3 The method is not performed by any analyst until a Demonstration of Capability (DOC) is completed. Every analyst who performs this method has demonstrated acceptable accuracy and precision by passing a Demonstration of Capability study.

### 20.0 Equipment / Instrument Maintenance and Troubleshooting

20.1 The HRMS is under service contract with Waters, Inc. For any major problems, it is best to allow certified technicians to work on the instrument. Service contract #P608. Telephone 1-800-252-4752

20.2 Routine GC maintenance:

20.2.1 At least once a week or for every 100 injections, the capillary inlet inserts will be replaced. The septa will be checked at least every 100 injections or more often as necessary.

20.2.2 Chromatograms exhibiting peak tailing, low peak response of standards or breakdown products may indicate that immediate maintenance is needed.

20.2.3 Instructions

20.2.3.1 Allow the injection port to cool to room temperature and shut off the gas flows to the instrument. Remove the septa nut and replace the



- used septa with a new one. Replace the nut with hand tight plus  $\frac{1}{4}$  turn of a wrench.
- 20.2.3.2 Remove the injection port assembly using a wrench to loosen the nut. Take out the used glass inlet liner and replace it with a new one.
  - 20.2.3.3 Bake the GC for one hour using the following temperatures: Oven 290°C, injector ports 275°C, and detectors 350°C. Column maximum temperatures are listed in the documentation accompanying a new column.
  - 20.2.3.4 A continuing calibration check standard will be injected to verify the calibration curve. If continuing calibration fails to meet criteria as stated in the method, a complete linearity of standards applicable to each type of analysis will be injected.
- 20.3 Periodic GC maintenance:
- 20.3.1 Replacing split seal (frit) for capillary inlet systems:
    - 20.3.1.1 The split seal (frit) (fig. 18-3 HP ref. Manual II) is commonly contaminated when dirty samples are run through the GC. Reduced peak size or breakdown peaks may indicate cleaning of the split seal is needed.
    - 20.3.1.2 After turning GC temperatures down, remove the column from the injection port and unscrew the reducing nut.
    - 20.3.1.3 The split seal (frit) and flat washer located inside the reducing nut are removed and replaced.
    - 20.3.1.4 Re-install column and turn the oven temperature up to 290°C until the detector signal is stable.
  - 20.3.2 Cleaning injector ports:
    - 20.3.2.1 Turn off the oven and remove the analytical column when the oven has cooled.
    - 20.3.2.2 Lower the injection port temperature to room temperature.
    - 20.3.2.3 Remove the glass injection port insert.
    - 20.3.2.4 Place a beaker beneath the injector port inside the GC oven. Using a Teflon wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene, catching the rinsate in the beaker.
    - 20.3.2.5 Prepare a solution of deactivation agent (Sylon-CT or equivalent) following manufacturer's directions. Coat all metal surfaces inside the injector body with the deactivation solution, then serially rinse the injector body with toluene, methanol, acetone and hexane.
    - 20.3.2.6 Reassemble the injector and replace the GC column.
  - 20.3.3 Cutting the Column:
    - 20.3.3.1 With continued sample analysis, contaminants tend to build up in the injection port side of the capillary column, causing decreased peak response and peak tailing.
    - 20.3.3.2 Using a wrench, remove the column inlet nut from inside the GC oven. Pull the column out of the nut and remove the graphite ferrule within the nut.
    - 20.3.3.3 Use a column scoring tool to remove 1 –2 loops from the inlet side of the column and discard.
    - 20.3.3.4 Thread the inlet nut back onto the column, and then slide a new ferrule onto the column. Allow the column to protrude 6mm from the



top of the ferrule and replace back into the inlet using a wrench to tighten.

### 20.4 Annual Gas Line Purifier Replacement

20.4.1 Changing oxygen/moisture indicator tube (OMI-1): The OMI-1 tube, located downstream from the gas purifier, indicates potential contamination within the carrier gas by rate of color change in the tube.

20.4.1.1 Turn down GC temperatures.

20.4.1.2 Unplug the heated converter tube. Turn off gas flow to line.

20.4.1.3 Using the parts in the OMI-1 installation kit, slide end caps onto ends of gas line, then finger tighten reducing union bodies to line ends. Wrench tighten approximately 1¼ additional turns.

20.4.1.4 Carefully remove plastic end caps from new tube. Avoid damaging foil seals that protect tube contents from exposure to air. Seals will be punctured by piercing needles in reducing unions when the tube is connected to the system.

20.4.1.5 Place nuts and new ferrules on the tube. Push exit of the tube into the reducing body closest to the instrument. Screw the nut and ferrule onto the union body and finger tighten. The piercing needle will penetrate the foil seal as you push the tube into the union. Slide the tube holder over the OMI-1 tube and gas line until the tube inlet is exposed.

20.4.1.6 Push the inlet end of the tube into the reducing union body. Position a slotted washer for the tube holder over the junction in one reducing union. Place the end cap on the tube holder and tighten until snug. Repeat at other end of tube holder. Turn the gas back on.

20.4.1.7 DO NOT ATTEMPT TO REMOVE THE CONTENTS OR REUSE THE TUBE. Spent resin contains a strong alkali.

20.4.1.8 Any partially spent tube should be placed in a glass beaker and stored away from combustible materials until the resin is a uniform brown. Dispose of the tube as a hazardous solid waste in accordance with applicable federal, state and local regulations.

### 20.5 HR Mass Spec Maintenance

20.5.1 APPL Inc. maintains a service contract with the instrument manufacturer (Waters Inc). Due to the technical nature of the HRMS, only Waters Inc service engineers are allowed to perform maintenance on the following mass spec components: the source, the flight tube and the mass spec detector.

20.5.2 Place a service call to Waters Inc if the following are observed: decrease in detector response, problems calibrating the mass axis, decreased beam transmission, low resolution checks or standard calibration problems with the ICAL or CCVs.

## 21.0 Computer Hardware and Software

21.1 Waters Laboratory Informatics. MassLynx v4.1 SCN 714 Desktop. Copyright © 2009 Waters Inc. All rights reserved.

21.1.1 This software is used in both acquiring the data from the HRMS and for processing data.

21.1.2 Most questions can be answered by accessing the 'Help' section of the software.





# Standard Operating Procedure

## QA Control Copy #   3

SOP: HPL8290  
 Section: 10  
 Revision: 11  
 Date: 07/23/14

**TABLE 1**  
 Calibration Standard Concentrations and  
 Acceptance Criteria for CCV, LCS and IDP Study

ANALYTE	ICAL CS- 0.2 ng/m l	ICAL CS-1 ng/m l	ICAL CS-2 ng/m l	ICAL CS-3 ng/ml	ICAL CS-4 ng/m l	ICAL CS-5 ng/m l	CCV	IDC Study		LCS	Labeled C13 surroga te
								% D	% REC		
2,3,7,8-TCDD	0.1	0.50	2.0	10	40	200	20	70-130	70-130	*	
2,3,7,8-TCDF	0.1	0.50	2.0	10	40	200	20	70-130	70-130	*	
1,2,3,7,8-PeCDD	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,7,8-PeCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
2,3,4,7,8-PeCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,4,7,8-HxCDD	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,6,7,8-HxCDD	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,7,8,9-HxCDD	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,4,7,8-HxCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,6,7,8-HxCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,7,8,9-HxCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
2,3,4,6,7,8-HxCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,4,6,7,8-HpCDD	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,4,6,7,8-HpCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
1,2,3,4,7,8,9-HpCDF	0.50	2.5	10	50	200	1000	20	70-130	70-130	*	
OCDD	1.0	5.0	20	100	400	2000	20	70-130	70-130	*	
OCDF	1.0	5.0	20	100	400	2000	20	70-130	70-130	*	



**Labeled Surrogate Standards**

C <sub>13</sub> -2,3,7,8,-TCDD	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -2,3,7,8,-TCDF	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,7,8,- PeCDD	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,7,8,- PeCDF	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,6,7,8,- HxCDD	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,6,7,8,- HxCDF	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,4,6,7,8,- HpCDD	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -1,2,3,4,6,7,8,- HpCDF	100	100	100	100	100	100		30		40-135	*	40-135
C <sub>13</sub> -OCDD	200	200	200	200	200	200		30		40-135	*	40-135

**C13 Labeled Internal Standards**

C <sub>13</sub> -1,2,3,4-TCDD <sup>A</sup>	100	100	100	100	100	100		30		*	*	*
C <sub>13</sub> -1,2,3,7,8,9- HxCDD <sup>B</sup>	100	100	100	100	100	100		30		*	*	*

<sup>A</sup> Used to determine the recoveries of the TCDD, TCDF, PeCDD and PeCDF surrogates.

<sup>B</sup> Used to determine the recoveries of the HxCDD, HxCDF, HpCDD, HpCDF and OCDD surrogates



**TABLE 2**  
**GC Performance Check Standard**

Analyte	No. Cl Atoms	Concentration ng/ml
1,3,6,8-TCDD and -TCDF	4	80
1,2,8,9- TCDD and -TCDF	4	80
C <sub>13</sub> -2,3,7,8 –TCDD and -TCDF	4	80
2,3,7,8-TCDD and -TCDF	4	80
1,2,3,4 and 1,2,3,7-TCDD	4	80
1,2,3,8 and 1,2,3,9-TCDD	4	80
2,3,4,7 and 1,2,3,9-TCDF	4	80
1,2,4,6,8 and 1,2,4,7,9-PeCDD	5	80
1,3,4,6,8-PeCDF	5	80
1,2,3,8,9-PeCDD and -PeCDF	5	80
1,2,4,6,7,9 and 1,2,4,6,8,9-HxCDD	6	80
1,2,3,4,6,7-HxCDD	6	80
1,2,3,4,6,8 and 1,2,3,4,8,9-HxCDF	6	80
1,2,3,4,6,7,9-HpCDD	7	80
1,2,3,4,6,7,8-HpCDD and -HpCDF	7	80
1,2,3,4,7,8,9-HpCDF	7	80

**TABLE 3**  
**C<sub>13</sub> Internal Standards and Recovery Standards**

Analyte	Internal Standard Concentration ng/ml	Recovery Standard Concentration ng/ml
C <sub>13</sub> -2,3,7,8,-TCDD	40	----
C <sub>13</sub> -2,3,7,8,-TCDF	40	----
C <sub>13</sub> -1,2,3,7,8,-PeCDD	40	----
C <sub>13</sub> -1,2,3,7,8,-PeCDF	40	----
C <sub>13</sub> -1,2,3,6,7,8,-HxCDD	100	----
C <sub>13</sub> -1,2,3,4,7,8,-HxCDF	100	----
C <sub>13</sub> -1,2,3,4,6,7,8,-HpCDD	100	----
C <sub>13</sub> -1,2,3,4,6,7,8,-HpCDF	100	----
C <sub>13</sub> -OCDD	200	----
C <sub>13</sub> -1,2,3,4-TCDD	----	40
C <sub>13</sub> -1,2,3,7,8,9-HxCDD	----	40



**TABLE 4**  
**Dioxin and Furan Spike Mix**

Analyte	Concentration ng/ml
2,3,7,8-TCDD	100
2,3,7,8-TCDF	100
1,2,3,7,8-PeCDD	250
1,2,3,7,8-PeCDF	250
2,3,4,7,8-PeCDF	250
1,2,3,4,7,8-HxCDD	250
1,2,3,6,7,8-HxCDD	250
1,2,3,7,8,9-HxCDD	250
1,2,3,4,7,8-HxCDF	250
1,2,3,6,7,8-HxCDF	250
1,2,3,7,8,9-HxCDF	250
2,3,4,6,7,8-HxCDF	250
1,2,3,4,6,7,8-HpCDD	250
1,2,3,4,6,7,8-HpCDF	250
1,2,3,4,7,8,9-HpCDF	250
OCDD	500
OCDF	500

**TABLE 5**  
**Theoretical Ion Abundance Ratios and QC Limits**

Cl Atom	m/z Forming Ratio	Theoretical Ratio	Ratio Acceptance Range
4	$m/m+2$	0.77	0.65 – 0.89
5	$m/(m+2)$	1.55	1.32 - 1.78
6	$(m+2)/(m+4)$	1.24	1.05 – 1.43
6	$m/(m+2)$	0.51	0.43 – 0.51
7	$(m+2)/(m+4)$	1.04	0.88 – 1.20
7	$m/(m+2)$	0.44	0.37 – 0.051
8	$(m+2)/(m+4)$	0.89	0.76 – 1.02



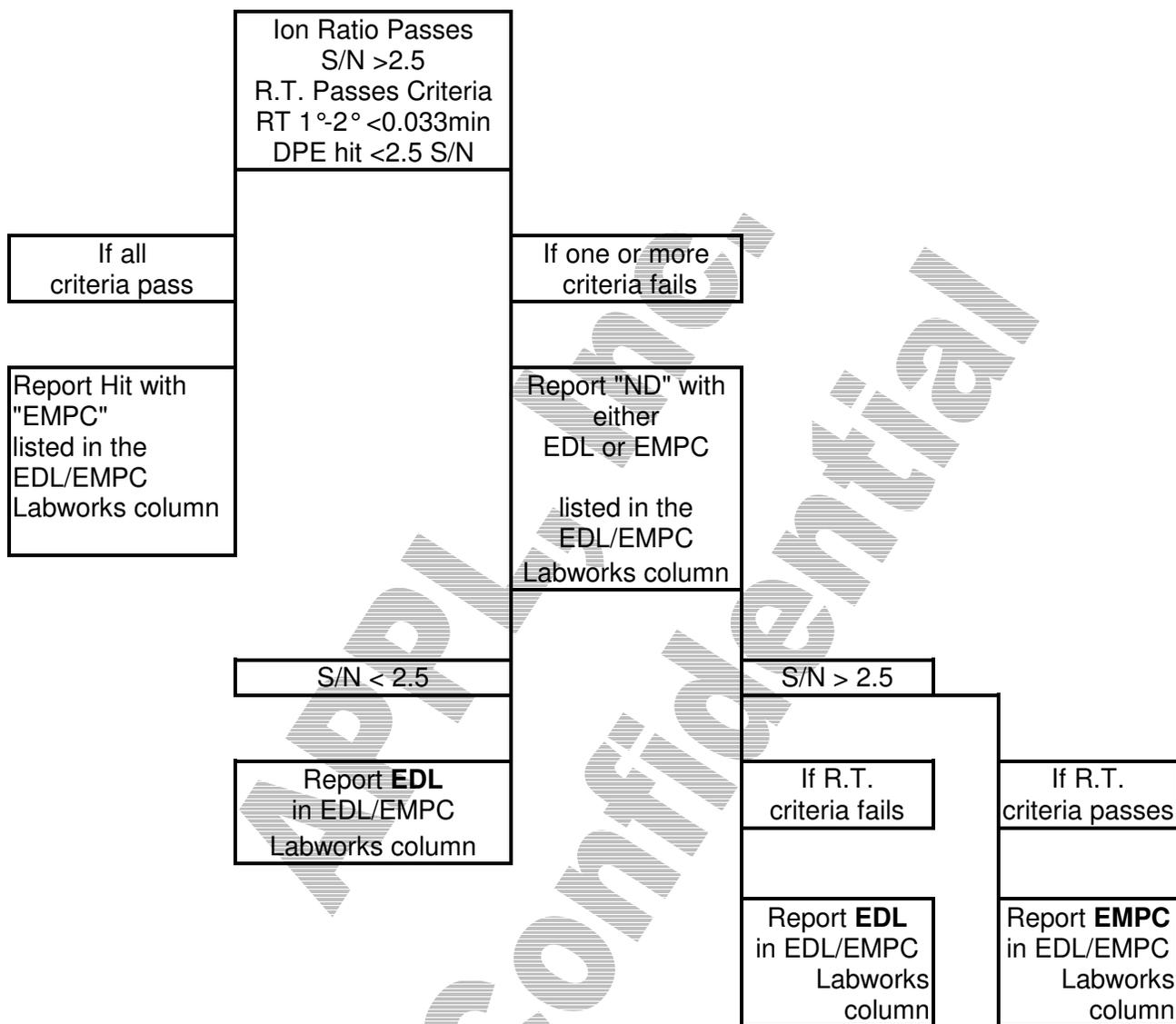
**TABLE 6**  
**2.3.7.8-TCDD Toxicity Equivalency Factors (TEFs)**

Analyte	1998 TEF	2005 TEF
2,3,7,8-TCDD	1	1
1,2,3,7,8-PeCDD	1	1
1,2,3,6,7,8-HxCDD	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1
1,2,3,4,7,8-HxCDD	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01
1,2,3,4,6,7,8,9-OCDD	0.001	0.003
2,3,7,8-TCDF	0.1	0.1
1,2,3,7,8-PeCDF	0.05	0.03
2,3,4,7,8-PeCDF	0.5	0.3
1,2,3,6,7,8-HxCDF	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1
1,2,3,4,7,8-HxCDF	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01
1,2,3,4,6,7,8,9-OCDF	0.001	0.003

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**TABLE 7**  
**Sample Reporting Criteria - Flow Chart**





## Standard Operating Procedure

TABLE 8

Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Resolving Power</b>	Prior to ICAL and at the beginning and the end of each 12-hour period of analysis.	Static resolving power $\geq$ 10,000 (10% valley) for identified masses.	Retune instrument and verify. Rerun affected samples.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
<b>Performance Check</b>	Prior to ICAL or calibration verification. At the beginning of each 12-hr period during which samples or calibration solutions are analyzed.	<p><u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers</u>: Resolved with a valley of <math>\leq</math> 25%.</p> <p>Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram.</p> <p>Absolute retention times for switching from one homologous series to the next <math>\geq</math> 10 sec. for all components of the mixture.</p>	Correct problem then repeat column performance check.	Flagging is not appropriate.	<p>Use GC column performance check solution if the laboratory operates during consecutive 12-hr periods.</p> <p>No samples shall be analyzed until performance check is within criteria.</p>
<b>Initial calibration (ICAL) for all analytes identified in method</b>	At instrument setup and after ICV or CCV failure, prior to sample analysis, and when a new lot is used as standard source for HRCC-3, sample fortification (IS), or recovery solutions.	<p>Ion abundance ratios in accordance with the method.</p> <p>S/N ratio <math>\geq</math> 10 for all reported analyte ions. RSD <math>\leq</math> 20% for the response factors (RF) for all 17 unlabeled standards. RSD <math>\leq</math> 20% for the RFs for the 9 labeled IS.</p>	Correct problem, then repeat ICAL.	Flagging is not appropriate.	<p>No samples shall be analyzed run until ICAL has passed.</p> <p>Calibration may not be forced through origin.</p>



# Standard Operating Procedure

## QA Control Copy #   3

SOP: HPL8290

Section: 10

Revision: 11

Date: 07/23/14

**TABLE 8, continued**

<b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b>					
<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Flagging Criteria</b>	<b>Comments</b>
<b>Initial Calibration Verification (ICV)</b>	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Ion abundance specified in the method must be met;. For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ D of the mean of RF established in ICAL.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
<b>Calibration Verification (CCV)</b>	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance specified in the method must be met. For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ D of RF established in ICAL.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.  <u>End-of-run CCV:</u> If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ RPD (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.





# Standard Operating Procedure

## QA Control Copy #   3

SOP: HPL8290  
 Section: 10  
 Revision: 11  
 Date: 07/23/14

**TABLE 8, continued**

<b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b>					
<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Flagging Criteria</b>	<b>Comments</b>
<b>Calibration Verification (CCV)</b>			CCVRFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new initial calibration if it is analyzed within two hours.  Otherwise analyze samples with positive detections, if necessary.		
<b>Internal Standards (IS)</b>	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to dilutions) must be within 40 – 135% of the ICAL average RF.	Correct problem, then re-prep and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples and explain in the case narrative.	
<b>Method Blank (MB)</b>	One per preparatory batch, run after calibration standards and before samples.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.



# Standard Operating Procedure

## QA Control Copy #   3

SOP: HPL8290

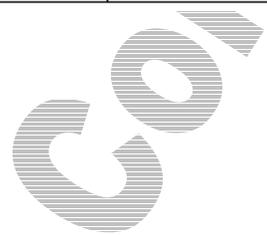
Section: 10

Revision: 11

Date: 07/23/14

**TABLE 8, continued**

<b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b>					
<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Flagging Criteria</b>	<b>Comments</b>
<b>Laboratory Control Sample (LCS)</b>	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.  If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported.  Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
<b>Matrix Spike (MS)</b>	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.  If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported.  If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
<b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b>	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.  If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.  MSD or MD: RPD of all analytes $\leq$ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.





**Standard Operating Procedure**

**QA Control Copy #   3**

SOP: HPL8290  
 Section: 10  
 Revision: 11  
 Date: 07/23/14

**TABLE 8, continued**

<b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b>					
<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Flagging Criteria</b>	<b>Comments</b>
<b>Internal Standards (IS)</b>	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to dilutions) must be within 40 – 135%.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples.	
<b>Sample Estimated Maximum Possible Concentration (EMPC)</b>	Every sample with a response S/N $\geq$ 2.5 for both quantitation ions.	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be $\geq$ 2.5.	NA.	Flagging is not appropriate.	
<b>Sample 2,3,7,8-TCDD toxicity equivalents (TEQ) concentration</b>	All positive detections.	Per method.	NA.	Flagging is not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.

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SOP REVIEW FORM

EMAX-218.6  
SOP No.

Rev. 5  
Revision Number

HEXAVALENT CHROMIUM  
Title

Document the review process and comment. Check the "NA" box if it is not applicable to the SOP, check the "Yes" box if you have fully understood the section, check "No" if you have any questions or if there are discrepancies with the current practice. Discuss the issue with the mentor/supervisor. If you need more space use the allocated space below or attach additional page.

SECTION	Yes	No	NA	COMMENTS
Scope & Application	✓			
Summary of Method	✓			
Detection Limits	✓			
Dynamic Range	✓			
Sample Holding Time & Preservation	✓			
Associated SOPs	✓			
Safety	✓			I have read all MSDS listed in this SOP
Instruments, Chemicals & Reagents	✓			
Standards	✓			
Procedures	✓			
- Sample Preparation	✓			
- Instrument Parameters	✓			
- Calibration	✓			
- Analysis	✓			
- Data Reduction	✓			
- Calculations	✓			
- Report Generation	✓			
- Data Review	✓			
- Preventive Maintenance	✓			
Quality Control	✓			
Corrective Action	✓			
Pollution Prevention	✓			
Waste Management	✓			
Supplementary Notes	✓			
References	✓			
Appendices	✓			

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This is to acknowledge that I have read, understood and agree to perform the procedures set forth in this SOP. Print your name & affix your signature.

Reviewed By

*[Signature]*  
LUCIA ARZANO

Date:

06/19/15

STANDARD OPERATING PROCEDURES  
**HEXAVALENT CHROMIUM**

SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

Prepared By: Lucita Arzadon *R. P. Arzadon* Date: 02-18-11

Approved By: Kenette Pimentel *K. Pimentel* Date: 02-18-11  
QA Manager

Approved By: Caspar Pang *C. Pang* Date: 02-18-11  
Laboratory Director

Control Number: 218.6-05-

### 1.0 SCOPE AND APPLICATION

- 1.1. This method is applicable for the determination of Hexavalent Chromium in groundwater, drinking water, and industrial wastewater effluents by Ion Chromatography. This SOP is an adaptation of USEPA Method 218.6.

### 2.0 SUMMARY OF METHOD

- 2.1. An aqueous sample is filtered through a 0.45- $\mu$ m filter and the filtrate is adjusted to a pH of 9.0 to 9.5 with a buffer solution or 2.5% NaOH 50% (w/w). A measured volume of the sample (250-1000  $\mu$ L) is introduced into the ion chromatograph. A guard column removes organics from the sample before the Cr(VI) as  $\text{CrO}_4^{2-}$  is separated on an anion exchange separator column. Post-column derivatization of the Cr(VI) with diphenylcarbazide is followed by detection of the colored complex at 530 nm.

#### 2.2. Interferences

- 2.2.1. **Contamination** – A trace amount of Cr is sometimes found in reagent grade salts. Since a concentrated buffer solution is used in this method to adjust the pH of samples, reagent blanks should be analyzed to assess for potential Cr(VI) contamination. Contamination can also come from improperly cleaned glassware or contact or caustic or acidic reagents of samples with stainless steel or pigmented material.
- 2.2.2. Reduction of Cr(VI) to Cr(III) can occur in the presence of reducing species in an acidic medium. However, at a pH of 6.5 or greater,  $\text{CrO}_4^{2-}$ , which is less reactive than the  $\text{HCrO}_4^-$ , is the predominant species.
- 2.2.3. Overloading of the analytical column capacity with high concentrations of anionic species, especially chloride and sulfate, will cause a loss of Cr(VI). The column specified in this method can handle samples containing up to 5% sodium sulfate or 2% sodium chloride. Poor recoveries from fortified samples and tailing peaks are typical manifestations of column overload.

### 3.0 DETECTION LIMITS

#### 3.1. Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

- 3.1.1. Refer to EMAX-QA04 for generation, validation and verification for DL, LOD and LOQ.
- 3.1.2. Established limits are:

MATRIX	DL	LOD	LOQ
Water ( $\mu\text{g/L}$ )	0.05	0.1	0.20

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

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**4.0 DYNAMIC RANGE**

- 4.1. Dynamic range is bracketed by the lowest and the highest calibration point, otherwise known as the calibration range.
- 4.2. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.3). All samples analyzed above this range shall be considered "over-range" and shall require dilution for proper quantitation.
- 4.3. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range shall be considered as "under-range" and shall require lower dilution factor for proper quantitation.

**5.0 SAMPLE HOLDING TIME AND PRESERVATION**

- 5.1. Aqueous samples shall be collected in either HPDE or glass bottles. They shall be cooled and stored at  $\leq 6^{\circ}\text{C}$  without freezing after collection until analysis is completed.
- 5.2. Unpreserved samples must be analyzed within 24 hours from the time of sample collection.
- 5.3. To extend the holding time to 28 days after sample collection, samples have to be preserved with ammonium sulfate or 2.5% NaOH 50% (w/w) to pH 9.3-9.7.

**6.0 ASSOCIATED SOPs**

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA04 Method Detection Limit Study
- 6.3. EMAX-QA08 Corrective Action
- 6.4. EMAX-QC02 Analytical Standard Preparation
- 6.5. EMAX-SM04 Analytical and QC Labeling

**7.0 SAFETY**

- 7.1. Read all MSDS for chemicals listed in this SOP.
- 7.2. All reagents, standards and samples shall be treated as potential hazards. Observe the standard laboratory safety procedures. Protective gear, i.e. lab coat safety glasses, gloves, shall be worn at all times when performing this procedure.
- 7.3. All waste generated during this analytical process shall be placed in waste containers. These wastes shall be endorsed to the waste disposal unit for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with copious amounts of tap water. If irritations persist, inform your supervisor immediately so proper action can be taken.

**8.0 INSTRUMENTS, CHEMICALS AND REAGENTS**

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11**8.1. Instrument**

IC	Dionex AD25/LC20 Chromatography and Dionex ICS-1000 IC
Guard Column	Dionex Ion Pack or equivalent
Column	Dionex Ion Pack AS7
Detector	UV detector
Autosampler	Dionex: AS40 Automated Sampler & AS50 Autosampler

**8.2. Supplies**

Autosampler vials	5 mL with filter caps; Vial kit: 1.5 mL with Slot Septum
Filters	0.45 $\mu$ m Gelman IC Acrodisc 4485 or equivalent
Volumetric Flasks	100, 250, 1000 mL
Containers	125 mL plastic snap seal
Micropipettes	1 and 5 mL; 10 and 200 $\mu$ L

**8.3. Chemicals**

Reagent water	Deionized water (ASTM Type II) or equivalent volume of 4 liters with reagent water.
Buffer Solution	Dissolve 165 g ammonium sulfate in 250 mL reagent water and add 32.5 mL ammonium hydroxide. Dilute to 500 mL with reagent water OR 25%NaOH 50% (w/w): Add 6.25g NaOH 50%(w/w) into 125 mL reagent water. Dilute to 250 mL.
Eluent solution	Dissolve 33 g of ammonium sulfate in 500 mL of reagent water; add 6.5 mL ammonium hydroxide. Dilute to 1 liter.
Post Column Reagent	Dissolved 0.5 g of 1,5-diphenylcarbazide in 100 mL of HPLC grade methanol in a 100-mL (4 oz) snap seal container. Add 28 mL of 98% sulfuric acid into 500 mL of reagent water, mix and degas with Nitrogen gas for 5 to 10 minutes prior to adding to 1,5-diphenylcarbazide solution. Dilute to 1 liter. Reagent is stable for four to five days.
Gas	N <sub>2</sub> Gas, high purity grade

**9.0 STANDARDS****9.1. Standard Preparation**

9.1.1. Refer to EMAX-QC02 for proper preparation of analytical standards.

9.1.2. Store all standard solutions at  $\leq 6^{\circ}\text{C}$ .**9.2. Stock Standards Solution (SSS)**9.2.1. **Primary Stock Standards** are purchased as neat standards from which a 1000-mg/L solution is prepared. These standards are primarily used for calibration.

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

9.2.2. **Secondary Stock Standards** are purchased commercially at 1000 mg/L as certified solutions from a different source. This standard is use to prepare ICV and QCS.

9.2.3. **Preparation of Intermediate Standard**

9.2.3.1. Intermediate standard, 1.0 mg/L. Dilute 100 µL of stock solution to 100 mL with the eluent.

9.2.3.2. Store all stock standards at 4°C.

9.3. **Calibration Standards (CAL)**

9.3.1. For initial calibration, prepare a minimum of six standards and a blank. Using a micropipette, add the intermediate standard solution (1.0mg/L), as suggested in the table below and dilute with the eluent to a final volume of 100 mL. Analyze the initial calibration standards prepared depending on the sensitivity of the instrument.

CAL Standard	Stock Standard Volume (µL)	Final Analyte Concentration (µg/L)
S0	0	0
S1	20	0.2
S2	200	2
S3	300	3
S4	500	5
S5	750	7.5
S6	1000	10

***NOTE: Final analyte concentration of the calibration points may vary depending upon the sensitivity of the instrument.***

9.4. **Quality Control Sample (QCS)**

9.4.1. Use the secondary stock solution to prepare QCS. This solution is used to spike ICV between the level of S4 & S5.

9.5. **Instrument Performance Check Standard (IPC)**

9.5.1. Use the primary stock standard for IPC at a spike level equivalent to S2. This solution is used for initial calibration check (ICC), continuing calibration check (CCC) and ending calibration check (ECC).

9.6. **Fortifying Standard (LFB/LFM)**

9.6.1. Prepare a working standard to fortify LFB and LFM from the stock standard, as described in 9.2, and diluted to a final volume of 100 mL with reagent water. Final concentration is 1 mg/mL.

10.0 **PROCEDURES**

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11**10.1. Sample Preparation**

- 10.1.1. Withdraw the samples from the sample control room and allow the samples to equilibrate to room temperature.
- 10.1.2. Using a 5-mL plastic syringe, withdraw 10 mL of sample and attach the 0.45- $\mu$ m filter. Discard the first 2 mL and collect the rest on a properly labeled sample container.
- 10.1.3. Repeat step 10.1.3 for all samples. Do the same for the LRB and LFB using the eluent.
- 10.1.4. Take clean sample vials equal to the number of samples to be analyzed not to exceed 20 field samples. If number of samples is  $\leq 10$  add vials for IPCs, LRB, LFB, and LFM; if samples  $> 10$  add three more vials for CCC, CCB, and LFM.
- 10.1.5. Spike 10  $\mu$ L of LFM standard to the LFB and LFM designated vials.
- 10.1.6. Using a calibrated micropipette, add 5 mL of sample to the sample vials, eluent for LRB and LFB, and the designated matrix spike sample for LFM.
- 10.1.7. Seal the vials and shake the QC samples to attain homogeneity of the mixture.

**10.2. Instrument Parameters**

Instrument	Dionex AD-25/ LC20 Chromatography; Dionex ICS-1000 IC
Detector	UV
Sample Flow Rate	1.0 ml/min
Isocratic Pump Pressure	900 – 2000
Sample Loop	250 $\mu$ l; 1 ml
Eluent Pressure	6-9 psi
Regenerant Pressure	55-70 psi
Run Time	10 min ; 14 min
Scale Setting ( $\mu$ S)	0.05 $\mu$ S

**10.2.1. Retention Time Window (RTW)****10.2.1.1. Establishing RTW**

- 10.2.1.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.
- 10.2.1.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained.
- 10.2.1.1.3. The width of RTW is defined by  $\pm 3X$  SD obtained from 10.2.1.1.2.

**10.2.1.2. Evaluating RTW**

- 10.2.1.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained. In the absence of previous study default to  $\pm 0.1$  minutes until a RTW is obtained.

**10.2.1.3. Application of RTW**

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

10.2.1.3.1. Establish the center of absolute retention time from the daily calibration check at the beginning of the analytical shift then apply the established RTW.

10.2.1.3.2. Whenever the observed retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analysis.

## 10.2.1.4. Updating RTW

10.2.1.4.1. Re-establish the RTW as described in Section 10.2.2.1 when any of the following condition occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacement of detector or column; temperature program change, etc.)

## 10.3. Calibration

## 10.3.1. Instrument Set Up

10.3.2. Set up the Dionex AD25/LC20 with the proper operating parameters established in Section 10.2.

10.3.2.1. Prime the pump to eliminate air bubbles in the system.

10.3.2.2. Start the flow of the regenerant by pressurizing a regenerant reservoir with Nitrogen to ensure constant delivery to the column.

10.3.2.3. Equilibrate the column for 30 minutes or until stable baseline is obtained.

## 10.3.3. Calibration (CAL)

10.3.3.1. Using the Chromeleon data acquisition program in the browser window, open the previous sequence and "Save as" under a new name. Go to the top square in the browser window and rename the method name. Sequence file display is the similar to the table below.

Sequence: IA12  
Operator: EMAXLABS

Page 1 of 1  
Printed: 1/15/2009 9:31:59 AM

Title: Temporary sequence for manual data acquisition

Datasource: DG7B3Q91\_local  
Location: DX60012009  
Timebase: DX600  
#Samples: 10

Created: 1/15/2009 9:26:42 AM by EMAXLABS  
Last Update: 1/15/2009 9:27:16 AM by EMAXLABS

No.	Sequence	Sample ID	Name	Type	Inj. Vol.	Program	Method	Status	Inj. Date/Time	Dil. Factor	Comment
1	IA12	001	IB	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
2	IA12	002	S-0	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
3	IA12	003	S-1	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
4	IA12	004	S-2	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
5	IA12	005	S-3	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
6	IA12	006	S-4	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
7	IA12	007	S-5	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
8	IA12	008	S-6	Standard	250.0	Cr6 Program	IC59A12	Single		1.0000	
9	IA12	009	ICV	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	
10	IA12	010	ICB	Unknown	250.0	Cr6 Program	IC59A12	Single		1.0000	

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

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- 10.3.3.2. Set the calibration standards on the Calibration Table as shown in Section 9.3.1.
  - 10.3.3.3. Place the standards chronologically into the Autosampler rack starting from Instrument Blank (IB).
  - 10.3.3.4. Go to Browser / Batch and start to initiate analysis of the standards.
  - 10.3.3.5. After all the standards are analyzed, plot the calibration curve of peak area against concentration and automatically it calculates the correlation coefficient ( $r^2$ ). Go to Report / Calibration (Current Peak) for the calibration curve and the  $r^2$ .
  - 10.3.3.6. Check Appendix 1 for acceptance criteria and corrective action.
  - 10.3.4. **Quality Control Sample (QCS)**
    - 10.3.4.1. Analyze ICV spiked with QCS after the initial calibration to verify the validity of the initial calibration and every 3 months thereafter.
  - 10.3.5. **Instrument Performance Check (IPC)**
    - 10.3.5.1. To verify the calibration curve, perform IPC.
    - 10.3.5.2. Analyze initial calibration check standard and initial calibration blank (ICC/ICB) at the beginning of the 24-hour shift.
    - 10.3.5.3. Analyze continuing calibration check standard (CCC) after every 10 samples or whenever the eluent is changed.
    - 10.3.5.4. Analyze ending calibration check standard (ECC) at the end of each analytical sequence of a 24-hour shift.
  - 10.4. **Analysis**
    - 10.4.1. **Analytical Sequence**
      - 10.4.1.1. ICC – initial calibration check
      - 10.4.1.2. ICB – initial calibration blank (reagent water)
      - 10.4.1.3. LRB – lab reagent blank
      - 10.4.1.4. LFB – lab fortified blank
      - 10.4.1.5. LFM – lab fortified matrix sample
      - 10.4.1.6. Samples – maximum of 10 field samples
      - 10.4.1.7. CCC – continuing calibration check
      - 10.4.1.8. LFM – another lab fortified matrix sample
      - 10.4.1.9. Samples – maximum of 10 field samples
      - 10.4.1.10. ECC – ending calibration check
    - 10.4.2. **Sample Result Evaluation**
      - 10.4.2.1. All sample runs, including the Laboratory Reagent Blank (LRB), Laboratory Fortified Blank Sample (LFB), Duplicate Sample (Dup), and Laboratory Fortified Sample Matrix (LFM), should be bracketed with calibration checks.
      - 10.4.2.2. Check QC results as soon as possible.

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- 10.4.2.3. If any analyte concentration exceeds the initial calibration range, perform appropriate dilution to bring the concentration to be within the range and reanalyze the dilution. All re-analyses due to dilution shall be bracketed with continuing calibrations.
- 10.4.2.4. Check each of the instrument performance checks that it meets the acceptance criteria set forth in Appendix 1.
- 10.4.2.5. Check that the retention time for all positive results falls within the established RTW.
- 10.4.2.6. Check the peaks for all positive results. Refer to Figure 1 for typical peak evaluation.
- The same peak integration technique applied in the initial calibration must be applied during the analysis of field samples.
  - Peaks must be well-resolved and properly integrated.
  - For manual integration refer to EMAX-DM01 (see section manual integration).
  - If a peak appears to be cryptic / anomalous, consult the supervisor.
- 10.4.2.7. Check if any of the sample results exceeds the calibration range. If such results exist, check that the diluted sample or extract is within the calibration range.
- 10.4.2.8. Rule-out any suspicion of carry-over. Any sample with trace amount of analyte(s) seen in a previous sample that exceeds the calibration range needs to be re-analyzed.
- 10.4.2.9. Check the LRB for absence or presence of contamination.
- 10.4.2.10. Check the LFB for method performance.
- 10.4.2.11. Check the LFM for absence or presence of matrix interference.

**10.5. Data Reduction**

- 10.5.1. Make a copy of the analytical run log and highlight the data to be reported.
- 10.5.2. Collect the reportable raw data separating the QC results from the sample results.
- 10.5.3. Keep all other data generated with the analytical folder marked with "For record only".

**10.6. Calculations**

- 10.6.1. For water samples, if the initial sample taken was V<sub>1</sub> and diluted to V<sub>2</sub> mL, then calculate the concentration using the following equation:

$$C_w = (C_i)(DF) \quad \text{Eq-10.6.1}$$

where:

- $C_i$  - Computer generated concentration in diluted digestate, in mg/L
- $C_w$  - Concentration in original sample, in mg/L
- $DF$  - Dilution factor =  $V_2/V_1$
- $V_1$  - Initial volume of the diluted sample, in mL

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**HEXAVALENT CHROMIUM**SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11 $V_2$  - Final volume of diluted samples, in mL10.6.2. Calculate for Percent Recovery of LFB

$$\% R = \left[ \frac{C_f}{C_o} \right] 100 \quad \text{Eq. 10.6.2}$$

where:

%R - Percent Recovery

 $C_f$  - Concentration found in LFB $C_o$  - Known Concentration of spiked solution10.6.3. Calculate for Percent Recovery of LFM

$$\% R = \left[ \frac{C_f - C_s}{C_o} \right] * 100 \quad \text{Eq. 10.6.3}$$

where:

%R - Percent Recovery

 $C_f$  - Concentration found in LFM $C_s$  - Concentration of the sample $C_o$  - Known Concentration of spiked solution10.6.4. Calculate for Precision

$$RPD = \frac{|C_1 - C_2|}{\left( \frac{C_1 + C_2}{2} \right)} * 100 \quad \text{Eq. 10.6.4}$$

where:

RPD - Relative Percent Difference

 $C_1$  - Concentration of the first measurement $C_2$  - Concentration of the second measurement10.6.5. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq. 10.6.5}$$

where:

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$SD$	-	<i>is the standard deviation</i>
$x_i$	-	<i>is the result at the <math>i^{th}</math> measurement</i>
$\bar{x}$	-	<i>is the mean</i>
$n$	-	<i>is the number of measurements</i>

**10.7. Report Generation**

- 10.7.1. Generate Form 1 to contain the sample results using WDBX<sup>1</sup>.exe and F1VX<sup>1</sup>.exe in series.
- 10.7.2. Generate Form 3 to contain the summaries of LFB and LFM and Sample Duplicate using IQCVX<sup>1</sup>.exe.
- 10.7.3. Generate the case narrative using CNX<sup>1</sup>.exe.
- 10.7.4. Assemble the analytical report in the order listed below.
  - 10.7.4.1. Case Narrative
  - 10.7.4.2. Lab Chronicle
  - 10.7.4.3. Sample Results [Form 1, raw data]
  - 10.7.4.4. QC Results [LRB, LFB, LFM each with raw data]
  - 10.7.4.5. Calibration [CAL, ICV, IPC, each with raw data]
  - 10.7.4.6. Analytical Log
  - 10.7.4.7. Sample Preparation Log
  - 10.7.4.8. Non-Conformance Report (if any)
- 10.7.5. Submit the analysis package for secondary review.

**10.8. Data Review****10.8.1. Check QC Criteria**

- 10.8.1.1. Check the analytical log that samples are analyzed in conformance to the QC frequency and all pertinent records are logged.
- 10.8.1.2. Check that the following conform to the QC requirement.
  - Holding Time
  - Calibrations
  - LRB
  - LFB
  - LFM

**10.8.2. Check Qualitative Identification**

- 10.8.2.1. Check the established RTW for the analytical batch that it was done properly.
- 10.8.2.2. Check that positively identified peaks are integrated properly and within the RTW.

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<sup>1</sup> X represents the latest version of the executable file.

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10.8.2.3. Check that suspicion of carry-over (if any) was ruled out.

**10.8.3. Check Quantitation**

10.8.3.1. Since a program generates the sample result forms, check the calculation of one sample result for correctness.

10.8.3.2. Check that dilution factors are properly factored in the calculation.

10.8.3.3. Check that correct sample amount and/or extract amount is properly factored.

**10.8.4. Check For Completeness**

10.8.4.1. Check that all Forms are present with their corresponding raw data.

10.8.4.2. Check that the case narrative accurately describes what transpired in the analytical process.

**10.9. Preventive Maintenance**

10.9.1. Perform daily instrument check prior to sample analysis. Refer to FORM 218.6FM – Instrument Maintenance Log.

<b>Maintenance Activity</b>	<b>Description</b>	<b>Frequency</b>
Verification	Prime system and run test injection Clean and inspect sampler and perform pressure test	Daily prior to analysis
Detector Maintenance	Inspect flow cell for leaks and verify performance	Daily prior to analysis
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
LC Pump Maintenance	Replace pump head seal, purge valve seal, and filter assembly frits. Perform wear-in procedure and leak test	Every six months or as necessary
Column Maintenance	Replace column switching valve rotor seal as necessary. Perform pressure test.	As necessary
Valve Maintenance	Replace rotor seal. Inspect valve fittings and capillaries for leaks	As necessary
System Cleaning	Remove dust from fans and vent covers	Every 6 months or as necessary
Sampler Maintenance	Replace rotor seal and needle seat assembly.	Once a year or as necessary
Inspection	Perform general inspection of the complete system	Once a year

10.9.2. Maintain an inventory of instrument parts and supplies for routinely maintenance.

**11.0 QUALITY CONTROL**

11.1. Initial Demonstration of Performance (IDP) shall be accomplished prior to implementation of this procedure and for each analyst the will perform the method. IDP shall constitute the successful completion of the following:

- Linear Calibration Range (LCR)
- Quality Control Sample (QCS)

## STANDARD OPERATING PROCEDURES

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- Method Detection Limit (MDL)
- 11.2. Assessing Laboratory Performance shall be demonstrated in every analytical batch and shall consist of complying to the requirement of the following:
- Lab Reagent Blank (LRB)
  - Lab Fortified Blank (LFB)
  - Instrument Performance Check (IPC)
- 11.3. Assessing Analyte Recovery Data Quality on a given matrix shall be demonstrated for each group of sample with similar matrix and shall consist of complying to the requirement of the following:
- Lab Fortified Sample Matrix Recovery (LFM)
  - LFM spike level must be high enough to be detected above the original sample and should not be less than 4 times the MDL.
  - If the concentration of fortification is less than 10% of the background concentrations measured in the unfortified sample, the matrix recovery should not be calculated.
  - If recovery for LFM falls outside the recovery range and the LFB is within control, the recovery problem for LFM is judged to be matrix related not system related.
- 11.4. Refer to Appendix 1 for all related Quality Control parameters, frequency, acceptance criteria and corrective action.

**12.0 CORRECTIVE ACTION**

- 12.1. Implement corrective action as described in Appendix 1.
- 12.2. **Sample Preparation QC**
- 12.2.1. For insufficient amount of sample, initiate a NCR and inform the PM immediately.
- 12.2.2. When laboratory reagent blank is non-compliant, investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.
- 12.2.3. If the analyte found in the laboratory reagent blank is not detected in any of the field samples, consult with the Supervisor and the PM if the result can be reported. Otherwise, re-analyze the method blank with the associated samples.
- 12.2.4. If the reagents that did not undergo quality control check were accidentally used, consider the following to correct the problem.
- 12.2.4.1. Check that there was no data integrity impact. Otherwise, repeat the analysis of all associated samples with new QC samples using QC'd reagent.
- 12.2.4.2. If method blank is clean, use the data to document the reagent QC.
- 12.3. **Sample Analysis QC**
- 12.3.1. When Instrument Performance Check (IPC) is non-compliant and all measures (e.g. flushing the column and/or changing the column, etc.) had been undertaken to correct the problem, consult the Supervisor for further advice prior to performing a new ICAL.
- 12.3.2. When flushing does not get rid of carry-over, consider changing the column.

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12.3.3. If the reagent water shows contamination in the instrument or in the laboratory reagent blank consider changing the filters of the reagent water source.

**12.4. Method QC**

12.4.1. When LOD verification is non-compliant, consider instrument maintenance and/or reestablish LOD. Refer to EMAX-QA04.

12.4.2. When retention time significantly shifts, check for any bubbles or leaks.

**12.5. Non-Conformance Report (NCR)**

12.5.1. Refer to EMAX-QA08 for details.

12.5.2. NCR is required when the following circumstances occur:

- Anomaly other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

**13.0 POLLUTION PREVENTION**

13.1. Quantity of chemicals purchased should be based on expected usage during its shelf life to minimize disposal of unused material.

13.2. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

13.3. Observe all necessary precautions to avoid spillage of reagents that may go to the wastewater drains.

**14.0 WASTE MANAGEMENT**

14.1. Collect all waste generated and properly turn them over to the waste disposal unit.

**15.0 SUPPLEMENTARY NOTES****15.1. Definition of Terms**

15.1.1. Analytical batch – is composed of a complete analysis for a batch of no more than 20 field samples. Every 10 field samples or a fraction thereof shall be bracketed with continuing calibration and one LFM is analyzed. For every analytical batch at least one LRB and one LFB is analyzed.

15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.3. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.4. Instrument Performance Check (IPC) – is a mid-range check standard containing the target analytes that is analyzed to verify the instrument calibration at the given criteria.

15.1.5. Laboratory Fortified Blank (LFB) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LFB is analyzed to monitor the accuracy of the analytical system.

## STANDARD OPERATING PROCEDURES

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- 15.1.6. Laboratory Fortified Sample Matrix (LFM) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. LFM is analyze to monitor matrix effect on a method’s recovery efficiency.
- 15.1.7. Laboratory Reagent Blank (LRB) – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.8. Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.
- 15.1.9. Matrix – is a component or form of a sample.
- 15.1.10. Quality Control Sample (QCS) – A solution obtained from a secondary source different from the source of calibration standard with known concentration of method analytes that is use to fortify an aliquot of ICV, LRB or LFM.
- 15.1.11. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.
- 15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.2. **Application of EMAX QC Procedures**
- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Hexavalent Chromium by Ion Chromatography analysis unless otherwise other directive is specified by the project requirements.
- 15.3. **Department of Defense (DoD) Projects**
- 15.3.1. Samples from DoD sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client’s quality control directive. In the absence of QAPP, the DoD Quality Systems Manual (QSM), latest update, shall be applied.
- 15.4. **Department of Energy (DoE) Projects**
- 15.4.1. Samples from DoE sponsored projects shall follow the Quality Assurance Project Plan (QAPP), Statement of Work (SOW) and/or client’s quality control directive. In the absence of QAPP, the DoE Quality Systems for Analytical Services (QSAS), latest update, shall be applied.
- 16.0 REFERENCES**
- 16.1. Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography, EPA Method 218.6 Rev. 3.3 (1994)
- 16.2. EMAX Quality Systems Manual, as updated.
- 17.0 APPENDICES**
- 17.1. **Figures**

## STANDARD OPERATING PROCEDURES

**HEXAVALENT CHROMIUM**

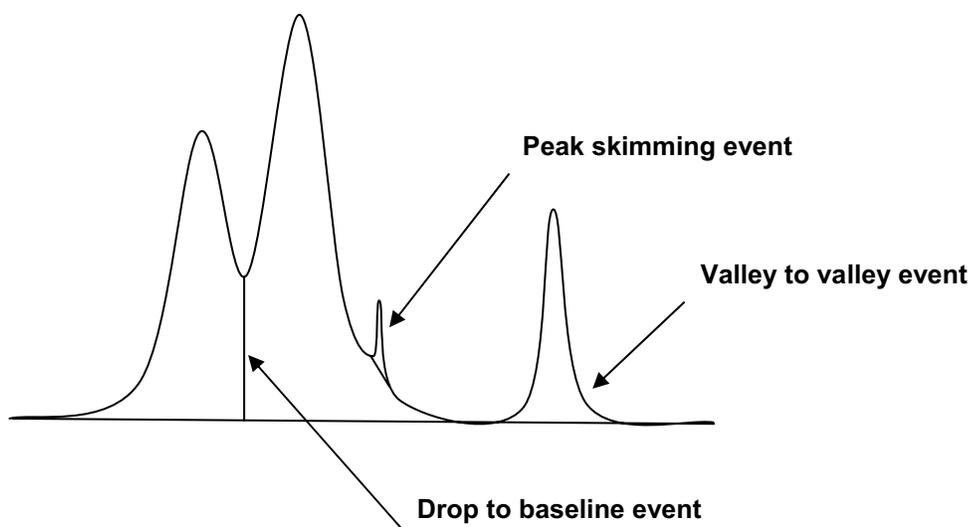
SOP No.: EMAX-218.6 Revision No. 5 Effective Date: 28-Feb-11

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- 17.1.1. Figure 1 Typical Peak Evaluation Technique
- 17.1.2. Figure 2 Typical Chromatogram
- 17.1.3. Figure 3 Typical ICAL Summary
- 17.1.4. Figure 4 Typical Sample Report
- 17.1.5. Figure 5 Typical LCS/LCSD Summary
- 17.1.6. Figure 6 Typical MS/MSD Summary
- 17.1.7. Figure 7 Typical Case Narrative
- 17.2. **Appendices**
  - 17.2.1. Appendix 1 Summary of Quality Control Procedures
  - 17.2.2. Appendix 2 Demonstration of Capability
- 17.3. **Forms**
  - 17.3.1. 218.6FA Analytical Run Log
  - 17.3.2. 218.6FS Sample Preservation Log
  - 17.3.3. 218.6FM Instrument Maintenance Log

**Figure 1: TYPICAL PEAK EVALUATION TECHNIQUE**

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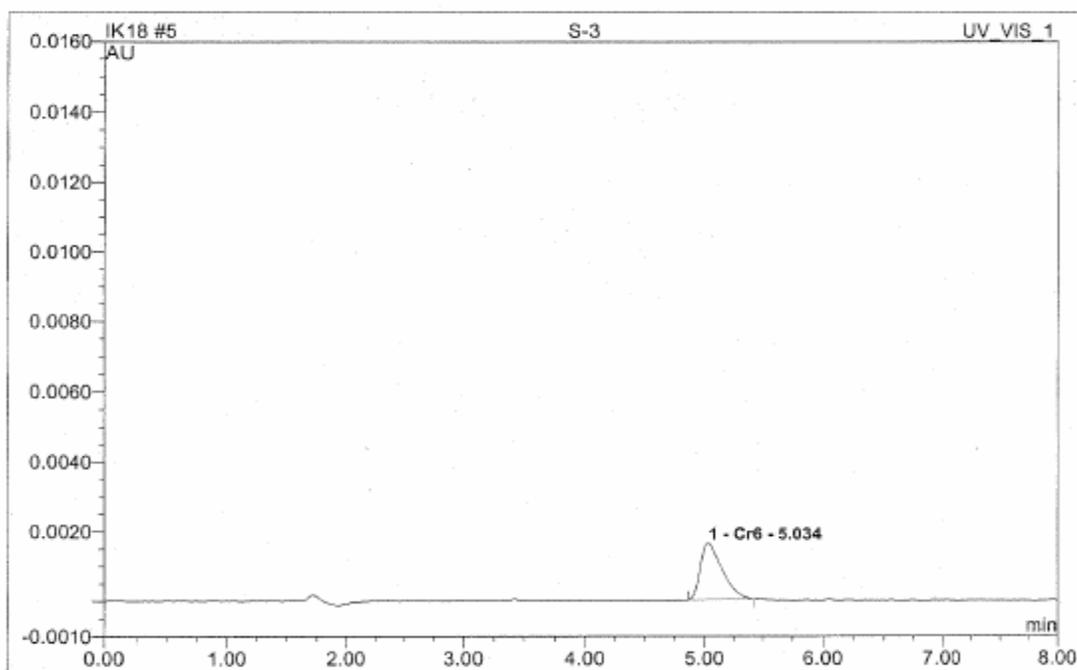


**Figure 2: TYPICAL CHROMATOGRAM**

Operator:EMAXLABS Timebase:DX600 Sequence:IK18

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<b>IK18 005 S-3</b>			
Sample Name:	S-3	Injection Volume:	250.0
Vial Number:	0	Channel:	UV_VIS_1
Sample Type:	standard	Wavelength:	n.a.
Control Program:	Cr6 Program	Bandwidth:	n.a.
Quantif. Method:	IC59K18	Dilution Factor:	1
Recording Time:	11/18/2009 12:52	Sample Weight:	1.0000
Run Time (min):	8.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height AU	Area AU*min	Rel.Area %	Amount	Type
1	5.03	Cr6	0.0016300	0.0003438	100.00	2.931	BMB
<b>Total:</b>			0.002	0.000	100.00	2.931	

*AS*  
 11/18/09

Report/Integration

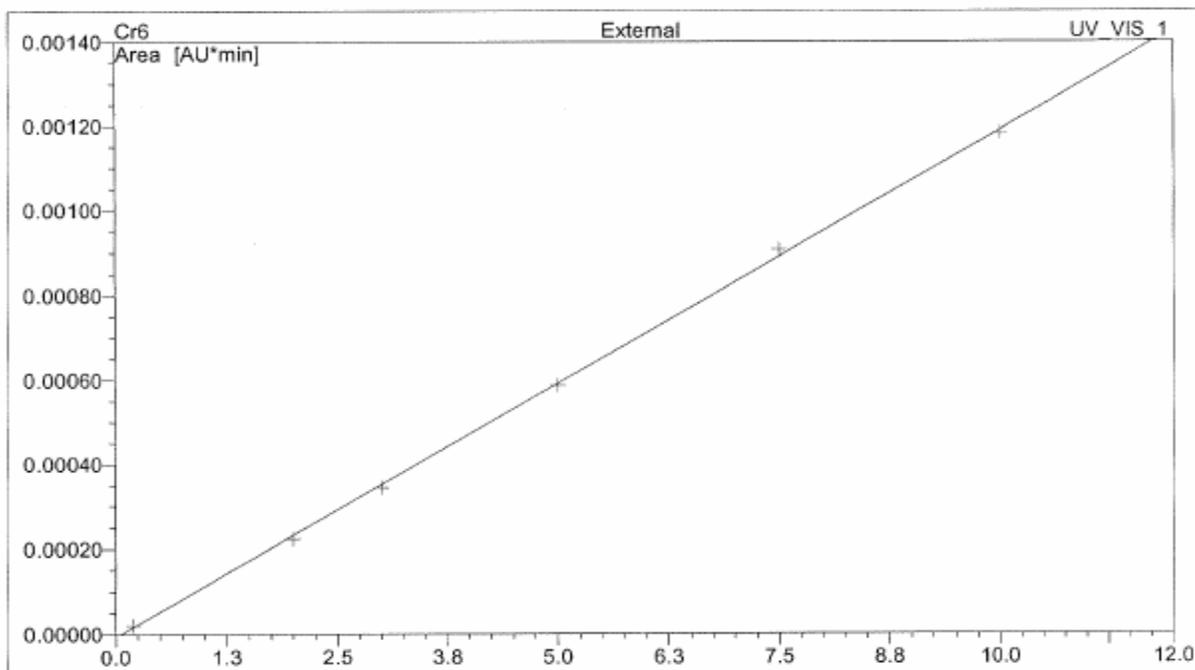
Chromeleon (c) Dionex 1996-2001  
 Version 6.70 SP2a Build 1871

**Figure 3: TYPICAL ICAL SUMMARY**

Operator:EMAXLABS Timebase:DX600 Sequence:IK18

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<b>8 S-6</b>			
Sample Name:	S-6	Injection Volume:	250.0
Vial Number:	0	Channel:	UV_VIS_1
Sample Type:	standard	Wavelength:	n.a.
Control Program:	Cr6 Program	Bandwidth:	n.a.
Quantif. Method:	IC59K18	Dilution Factor:	1.0000
Recording Time:	11/18/2009 13:23	Sample Weight:	1.0000
Run Time (min):	8.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Cal.Type	Points	R-Square	Offset	Slope	Curve
1	5.04	Cr6	0LOff	6	0.9995	-0.0000069	0.0001197	0.0000
<b>Average:</b>					0.9995	0.0000	0.0001	0.0000

*Handwritten signature and date: 11/18/09*

Figure 4:

## TYPICAL SAMPLE REPORT

METHOD 218.6  
HEXAVALENT CHROMIUM

Client : XYZ, INC  
 Project : CLEAN WATER PROJECT  
 Batch No. : 09L213

Matrix : WATER  
 Instrument ID : I59

SAMPLE ID	EMAX SAMPLE ID	RESULTS (ug/L)	DLF	MOIST	RL (ug/L)	MDL (ug/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
129178-6418	L213-05	ND	1	NA	0.200	0.100	12/10/0918:20	NA	IL10003	IL10001	HCL007W	12/10/0907:55	12/10/09
129178-6419	L213-06	ND	1	NA	0.200	0.100	12/10/0918:41	NA	IL10005	IL10001	HCL007W	12/10/0908:05	12/10/09
129178-7106	L213-11	ND	1	NA	0.200	0.100	12/10/0918:51	NA	IL10006	IL10001	HCL007W	12/10/0908:12	12/10/09
129178-7106MS	L213-11M	1.79	1	NA	0.200	0.100	12/10/0919:02	NA	IL10007	IL10001	HCL007W	12/10/0908:12	12/10/09
129178-7106MSD	L213-11S	1.71	1	NA	0.200	0.100	12/10/0919:12	NA	IL10008	IL10001	HCL007W	12/10/0908:12	12/10/09
129178-7104	L213-09	ND	1	NA	0.200	0.100	12/10/0919:54	NA	IL10011	IL10009	HCL007W	12/10/0908:48	12/10/09
LCS1W	HCL007WL	1.82	1	NA	0.200	0.100	12/10/0920:04	NA	IL10012	IL10009	HCL007W	NA	NA
LCD1W	HCL007WC	1.84	1	NA	0.200	0.100	12/10/0920:15	NA	IL10013	IL10009	HCL007W	NA	NA
MBLK1W	HCL007WB	ND	1	NA	0.200	0.100	12/10/0920:25	NA	IL10014	IL10009	HCL007W	NA	NA
129178-6416	L213-03	0.789	1	NA	0.200	0.100	12/10/0920:35	NA	IL10015	IL10009	HCL007W	12/10/0909:10	12/10/09
129178-6416MS	L213-03M	2.63	1	NA	0.200	0.100	12/10/0920:46	NA	IL10016	IL10009	HCL007W	12/10/0909:10	12/10/09
129178-6416MSD	L213-03S	2.68	1	NA	0.200	0.100	12/10/0920:56	NA	IL10017	IL10009	HCL007W	12/10/0909:10	12/10/09
129178-7105	L213-10	ND	1	NA	0.200	0.100	12/10/0921:07	NA	IL10018	IL10009	HCL007W	12/10/0909:11	12/10/09
129178-6420	L213-07	ND	1	NA	0.200	0.100	12/10/0921:27	NA	IL10020	IL10009	HCL007W	12/10/0912:00	12/10/09
129178-6417	L213-04	ND	1	NA	0.200	0.100	12/10/0922:09	NA	IL10023	IL10021	HCL007W	12/10/0908:35	12/10/09
129178-6415	L213-02R	0.293	1	NA	0.200	0.100	12/11/0909:15	NA	IL10028	IL10026	HCL007W	12/10/0910:50	12/10/09

**Figure 5: TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA  
 LCS/LCD ANALYSIS

CLIENT: XYZ, INC  
 PROJECT: CLEAN WATER PROJECT  
 BATCH NO.: 09L213  
 METHOD: METHOD 218.6

=====

MATRIX: WATER  
 DILUTION FACTOR: 1 1 1 % MOISTURE: NA  
 SAMPLE ID: MBLK1W  
 LAB SAMP ID: HCL007WB HCL007WL HCL007WC  
 LAB FILE ID: IL10014 IL10012 IL10013  
 DATE EXTRACTED: NA NA NA DATE COLLECTED: NA  
 DATE ANALYZED: 12/10/0920:25 12/10/0920:04 12/10/0920:15 DATE RECEIVED: NA  
 PREP. BATCH: HCL007W HCL007W HCL007W  
 CALIB. REF: IL10009 IL10009 IL10009

ACCESSION:

PARAMETER	BLNK RSLT (ug/L)	SPIKE AMT (ug/L)	BS RSLT (ug/L)	BS % REC	SPIKE AMT (ug/L)	BSD RSLT (ug/L)	BSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Hexavalent Chromium	ND	2.00	1.82	91	2.00	1.84	92	1	80-120	20

Figure 6:

TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA  
 MS/MSD ANALYSIS

CLIENT: XYZ, INC  
 PROJECT: CLEAN WATER  
 BATCH NO.: 09L213  
 METHOD: METHOD 218.6

MATRIX: WATER  
 DILUTION FACTOR: 1 1 1 % MOISTURE: NA  
 SAMPLE ID: 129178-6416  
 LAB SAMP ID: L213-03 L213-03M L213-03S  
 LAB FILE ID: IL10015 IL10016 IL10017  
 DATE EXTRACTED: NA NA NA DATE COLLECTED: 12/10/09 09:10  
 DATE ANALYZED: 12/10/0920:35 12/10/0920:46 12/10/0920:56 DATE RECEIVED: 12/10/09  
 PREP. BATCH: HCL007W HCL007W HCL007W  
 CALIB. REF: IL10009 IL10009 IL10009

ACCESSION:

PARAMETER	SMPL RSLT (ug/L)	SPIKE AMT (ug/L)	MS RSLT (ug/L)	MS % REC	SPIKE AMT (ug/L)	MSD RSLT (ug/L)	MSD % REC	RPD ( % )	QC LIMIT ( % )	MAX RPD ( % )
Hexavalent Chromium	0.789	2.00	2.63	92	2.00	2.68	94	2	75-125	20

Figure 7:

TYPICAL CASE NARRATIVE

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CASE NARRATIVE

Client : XYZ, INC

Project : CLEAN WATER PROJECT

SDG : 09L213

METHOD 218.6  
HEXAVALENT CHROMIUM

A total of nine (9) water samples were received on 12/10/09 for Chromium Hexavalent by IC analysis, Method 218.6 in accordance with the Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography, EPA Method 218.6 Rev. 3.3 (1994).

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Multi-calibration points were generated to establish initial calibration (ICAL). ICAL was verified using a secondary source. Continuing calibration verifications were carried out at the frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for HCL007WL/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at the frequency prescribed by the project. Percent recoveries for L213-03M/S were within project QC limits. Percent recoveries for L213-11M/S were within project QC limits.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

**Appendix 1:**

**SUMMARY OF QUALITY CONTROL PROCEDURES**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1 <sup>st</sup> Rvw	2 <sup>nd</sup> Rvw
Multipoint calibration for all analytes (CAL) [Min. 3 pts + Blank]	Every 6 months or when IPC fails to meet the acceptance criteria.	Correlation coefficient $\geq 0.999$ for linear regression	Correct the problem then repeat initial calibration		
Quality Control Sample (QCS/ICV)	After every initial calibration	All analytes within $\pm 10\%$ of expected value	Correct the problem then repeat initial calibration		
Instrument Performance Check (IPC/CCV)	Bracket every 10 field samples with IPC.	All analytes within $\pm 5\%$ of expected value	Repeat calibration and re-analyze all samples since last successful calibration		
Laboratory Reagent Blank (LRB)	One per preparation batch	No analytes detected $\geq$ LOQ	Re-prep and re-analyze LRB and all samples processed with LRB		
Laboratory Fortified Blank	One LFB per preparation batch	%R = 90 –110%	Re-prep and re-analyze the LCS and all associated samples		
Laboratory Fortified Matrix (LFM)	One LFM per 10 field samples per matrix	%R = 90 –110%	If LFB passed, no action		
Ending Calibration Check(ECC)	After each analytical sequence of a 24-hour shift	All analytes within $\pm 5\%$ of expected value	Correct the problem then repeat initial calibration and re-analyze all associated samples		
Method Detection Limit	Once every 6 months	None	None		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
			Date:		

**Appendix 2: DEMONSTRATION OF CAPABILITY**

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**DEMONSTRATION OF CAPABILITY  
 HEXAVALENT CHROMIUM  
 METHOD EPA 218.6**

Unit: µg/L  
 Instrument ID: 159

Date Analyzed: 07/06/10  
 Analyzed By: Andy Mai

PARAMETER	HCF015WL	HCF015WC	HCF016WL	HCF016WC	True Value	Ave. Conc.	Ave. % Rec.	SD	RSD	QC Criteria	COMMENTS
Hexavalent Chromium	1.925	1.988	1.995	2.038	2	1.99	99	0.047	2	80 - 120	Passed

218.6FA:

ANALYTICAL RUN LOG



ANALYSIS RUN LOG  
*for*  
 HEXAVALENT CHROMIUM IC

Page 1

Note: For samples and relevant QCs/Standards  
 analyzed, refer to attached analytical sequence.

Comments:

1,5 Diphenylcarbohydrozide : 0.5g  
 MeOH : 100 mL  
 H<sub>2</sub>SO<sub>4</sub> : 28 mL  
 (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> : 66g  
 NH<sub>4</sub>OH : 13 mL

Book #: A59-022

Instrument No.: 59

Analytical Sequence:

Method File:

Analytical Batch:

SOP #	Rev. #
<input type="checkbox"/> EMAX-7199	2
<input type="checkbox"/> EMAX-218.6	4
<input type="checkbox"/> EMAX-	

STANDARDS ID	
ICAL	
ICV	
CCV	
LCS	
MS	

ELECTRONIC DATA ARCHIVAL	
Location	Date
<input type="checkbox"/> CHROMELEON	
<input type="checkbox"/>	

Analyzed By: \_\_\_\_\_

Date: \_\_\_\_\_

218.6FS

SAMPLE PREPARATION LOG



SAMPLE PRESERVATION LOG FOR HEXAVALENT CHROMIUM IC

Page 1

EMAX-7199 Rev. #1

EMAX-218.6 Rev. #4

Book # PHC-004

Matrix:		Start Date	Time:	End Date	Time:			
Sample Prep ID	Lab Sample ID	Initial pH	Final pH	Notes	Standards	pH		
01					Buffer 7			
02					Buffer 10			
03					Check pH Buffer (8)			
04								
05								
06								
07					Reagent	Standard ID #		
08					NaOH			
09					(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> /NH <sub>4</sub> OH			
10								
11								
12					Legend:			
13					Color	Texture	Clarity	Artifacts
14					Bu = Blue Bl = Black	Cs = Coarse	Cr = Clear	Rk = Rocks
15					Bn = Brown Gn = Green	Md = Medium	Cy = Cloudy	Sl = Shale
16					Og = Orange Rd = Red	Fn = Fine	Td = turbid	Vg = Vegetation
17					Yw = Yellow			
18					Comments: _____			
19					_____			
20					_____			
21					_____			
22					_____			
23					Prepared By: _____			
24					Standard Added By: _____			
25					Checked By: _____			





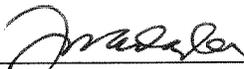
**LABORATORIES, INC.**  
1835 W. 205th Street  
Torrance, CA 90501  
Tel: (310) 618-8889  
Fax: (310) 618-0818

ADDENDUM TO

Document	ALL APPLICABLE ANALYTICAL AND SAMPLE PREPARATION SOPs
Revision Number	CURRENT REVISIONS
Section	10.0
Date	28 July 2014
Reference Number	AA.5

This applies to all sample preparation and analytical methods wherever filters are used.

Record the Lot Number of the filter used in the specific laboratory logbook. Where no specific location is provided, use the Comments Section of the log.

PREPARED BY:  Date: 07/28/14

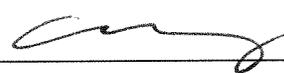
Name Farina Madamba

Title QA / QC Coordinator

APPROVED BY:  Date: 072814

Name Kenette Pimentel

Title QA Manager

APPROVED BY:  Date: 07-28-14

Name Caspar Pang

Title Laboratory Director

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**Attachment 8**  
**Laboratory Certifications/Accreditations**

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# PERRY JOHNSON LABORATORY ACCREDITATION, INC.

## Certificate of Accreditation

*Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:*

***APPL, Inc.***

***908 N. Temperance Avenue, Clovis, CA 93611***

*(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 “General Requirements for the competence of Testing and Calibration Laboratories” and the DoD Quality Systems Manual for Environmental Laboratories Version 5.0 July 2013 and is accredited in accordance with the:*

### **United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP)**

***This accreditation demonstrates technical competence for the defined scope:  
Environmental Testing  
(As detailed in the supplement)***

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body’s duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen  
President/Operations Manager

*Initial Accreditation Date:*

May 13, 2013

*Issue Date:*

November 28, 2013

*Revision Date:*

January 16, 2015

*Expiration Date:*

November 27, 2015

*Accreditation No.:*

74807

*Certificate No.:*

L13-238-R2

Perry Johnson Laboratory  
Accreditation, Inc. (PJLA)  
755 W. Big Beaver, Suite 1325  
Troy, Michigan 48084

*The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: [www.pjllabs.com](http://www.pjllabs.com)*



# Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

## APPL, Inc.

908 N. Temperance Avenue, Clovis, CA 93611  
Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

Matrix	Standard/ Method	Technology	Analyte
Aqueous	EPA 218.6	Ion Chromatography (IC)	Chromium VI
Aqueous	EPA 245.1	AAS	Mercury
Aqueous	EPA 7470A	AAS	Mercury
Aqueous	EPA 8011	GC/ECD	1,2,3-Trichloropropane
Aqueous	EPA 8011	GC/ECD	1,2-Dibromo-3-chloropropane (DBCP)
Aqueous	EPA 8011	GC/ECD	1,2-Dibromomethane (EDB, Ethylene dibromide)
Aqueous	EPA 9060A	Nondispersive Infrared Detector (NDIR)	Dissolved Organic Carbon
Aqueous	EPA 9060A	Nondispersive Infrared Detector (NDIR)	Total Organic Carbon
Aqueous	RSK-175	GC/FIC	Ethane
Aqueous	RSK-175	GC/FIC	Ethene
Aqueous	RSK-175	GC/FIC	Methane
Aqueous	SM 2320B	Titrimetric	Bicarbonate
Aqueous	SM 2320B	Titrimetric	Carbonate
Aqueous	SM 2320B	Titrimetric	Hydroxide
Aqueous	SM 2320B	Titrimetric	Total Alkalinity (CaCO <sub>3</sub> )
Aqueous	SM 2510B	EC Meter	Specific conductance, Conductivity (25C)
Aqueous	SM 2540C	Gravimetric	Total Dissolved Solids (TDS)
Aqueous	SM 2540D	Gravimetric	Non-Filterable Residue (TSS)
Aqueous	SM 4500-S2 F	Titrimetric	Sulfide
Aqueous	SM 5310B	Nondispersive Infrared Detector (NDIR)	Dissolved Organic Carbon
Aqueous	SM 5310B	Nondispersive Infrared Detector (NDIR)	Total Organic Carbon
Aqueous	SM 5520B	Gravimetric	Oil & Grease
Aqueous	SM 5520-BF	Gravimetric	TRPH (Gravimetric)
Aqueous	SM 5540C	UV/Vis	MBAS
Aqueous	SM3500-Fe Bc	Spectrophotometric	Ferrous Iron
Aqueous	SM4500-S2 F	Spectrophotometric	Sulfide
Aqueous	SM5310B	Total Organic Carbon Analyzer	Dissolved Organic Carbon
Aqueous	SM5310B	Total Organic Carbon Analyzer	Total Organic Carbon
Aqueous	EPA 160.1	Gravimetric	Total Dissolved Solids (TDS)
Aqueous	EPA 1664A	Gravimetric	n-Hexane Extractable Material (O&G)
Aqueous	EPA 1664A	Gravimetric	TPH (SGT-HEM)



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Matrix	Standard/Method	Technology	Analyte
Solids	AK103	GC/FID	Residual Range Organics, C25-C36
Solids	EPA 1030	Manual	Ignitability
Solids	EPA 7471A,B	AAS	Mercury
Solids	EPA 8015B,C,D	GC/FID	RRO (Residual Range Organics)
Solids	EPA 9045C,D	Ion Selective Electrode	pH/Corrosivity
Solids	WALKLEY-BLACK	Titration	Total Organic Carbon (TOC)
Aqueous/Solids	AK101	GC-FID	Gasoline Range Organics, C6-C10
Aqueous/Solids	AK102	GC-FID	Diesel Range Organics, C10-C25
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,3',4,4',5'-Pentachlorobiphenyl (PCB 123)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	2,4,4'-Trichlorobiphenyl (PCB 28)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	3,4,4',5-Tetrachlorobiphenyl (PCB 81)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (129)+(138)+(163)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (153)+(168)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (156)+(157)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (180)+(193)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (20)+(28)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCB (90)+(101)+(113)
Aqueous/Solids	EPA 1668A	High Res. GC/MS	PCBs, total
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Bromide
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Chloride



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Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Fluoride
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Nitrate as N (NO <sub>3</sub> - as N)
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Nitrite + Nitrate as N
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Nitrite as N
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Orthophosphate as P
Aqueous/Solids	EPA 300.0	Ion Chromatography (IC)	Sulfate (SO <sub>4</sub> )
Aqueous/Solids	EPA 350.1	Flow Injection Analysis (FIA)	Ammonia as N
Aqueous/Solids	EPA 351.2	Flow Injection Analysis (FIA)	Total Kheldahl Nitrogen
Aqueous/Solids	EPA 353.2	Flow Injection Analysis (FIA)	Nitrate as N (NO <sub>3</sub> as N)
Aqueous/Solids	EPA 353.2	Flow Injection Analysis (FIA)	Nitrate + Nitrate as N
Aqueous/Solids	EPA 353.2	Flow Injection Analysis (FIA)	Nitrite as N
Aqueous/Solids	EPA 6010B,C	ICP-OES	Aluminum
Aqueous/Solids	EPA 6010B,C	ICP-OES	Antimony
Aqueous/Solids	EPA 6010B,C	ICP-OES	Antimony
Aqueous/Solids	EPA 6010B,C	ICP-OES	Arsenic
Aqueous/Solids	EPA 6010B,C	ICP-OES	Arsenic
Aqueous/Solids	EPA 6010B,C	ICP-OES	Barium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Beryllium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Boron
Aqueous/Solids	EPA 6010B,C	ICP-OES	Cadmium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Calcium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Chromium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Cobalt
Aqueous/Solids	EPA 6010B,C	ICP-OES	Copper
Aqueous/Solids	EPA 6010B,C	ICP-OES	Iron
Aqueous/Solids	EPA 6010B,C	ICP-OES	Lead
Aqueous/Solids	EPA 6010B,C	ICP-OES	Magnesium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Manganese
Aqueous/Solids	EPA 6010B,C	ICP-OES	Molybdenum
Aqueous/Solids	EPA 6010B,C	ICP-OES	Nickel
Aqueous/Solids	EPA 6010B,C	ICP-OES	Potassium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Selenium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Silver
Aqueous/Solids	EPA 6010B,C	ICP-OES	Sodium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Strontium



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*Accreditation is granted to the facility to perform the following testing:*

<b>Matrix</b>	<b>Standard/Method</b>	<b>Technology</b>	<b>Analyte</b>
Aqueous/Solids	EPA 6010B,C	ICP-OES	Thallium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Tin
Aqueous/Solids	EPA 6010B,C	ICP-OES	Titanium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Total Phosphorus
Aqueous/Solids	EPA 6010B,C	ICP-OES	Vanadium
Aqueous/Solids	EPA 6010B,C	ICP-OES	Zinc
Aqueous/Solids	EPA 6020A	ICP-MS	Aluminum
Aqueous/Solids	EPA 6020A	ICP-MS	Antimony
Aqueous/Solids	EPA 6020A	ICP-MS	Arsenic
Aqueous/Solids	EPA 6020A	ICP-MS	Barium
Aqueous/Solids	EPA 6020A	ICP-MS	Beryllium
Aqueous/Solids	EPA 6020A	ICP-MS	Boron
Aqueous/Solids	EPA 6020A	ICP-MS	Cadmium
Aqueous/Solids	EPA 6020A	ICP-MS	Calcium
Aqueous/Solids	EPA 6020A	ICP-MS	Chromium
Aqueous/Solids	EPA 6020A	ICP-MS	Cobalt
Aqueous/Solids	EPA 6020A	ICP-MS	Copper
Aqueous/Solids	EPA 6020A	ICP-MS	Iron
Aqueous/Solids	EPA 6020A	ICP-MS	Lead
Aqueous/Solids	EPA 6020A	ICP-MS	Magnesium
Aqueous/Solids	EPA 6020A	ICP-MS	Manganese
Aqueous/Solids	EPA 6020A	ICP-MS	Molybdenum
Aqueous/Solids	EPA 6020A	ICP-MS	Nickel
Aqueous/Solids	EPA 6020A	ICP-MS	Potassium
Aqueous/Solids	EPA 6020A	ICP-MS	Selenium
Aqueous/Solids	EPA 6020A	ICP-MS	Silver
Aqueous/Solids	EPA 6020A	ICP-MS	Sodium
Aqueous/Solids	EPA 6020A	ICP-MS	Strontium
Aqueous/Solids	EPA 6020A	ICP-MS	Thallium
Aqueous/Solids	EPA 6020A	ICP-MS	Tin
Aqueous/Solids	EPA 6020A	ICP-MS	Titanium
Aqueous/Solids	EPA 6020A	ICP-MS	Vanadium
Aqueous/Solids	EPA 6020A	ICP-MS	Zinc
Aqueous/Solids	EPA 6850	HPLC/Electrospray Ionization/MS	Perchlorate
Aqueous/Solids	EPA 7196A	UV/Vis	Chromium VI



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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8015B,C,D	GC/FID	Diesel Range Organics
Aqueous/Solids	EPA 8015B,C,D	GC/FID	Gasoline Range Organics
Aqueous/Solids	EPA 8015B,C,D	GC/FID	Total Purgeable Hydrocarbons
Aqueous/Solids	EPA 8081A,B	GC/NPD	4,4'-DDD
Aqueous/Solids	EPA 8081A,B	GC/ECD	4,4'-DDE
Aqueous/Solids	EPA 8081A,B	GC/ECD	4,4'-DDT
Aqueous/Solids	EPA 8081A,B	GC/ECD	4,4'-Methoxychlor
Aqueous/Solids	EPA 8081A,B	GC/ECD	a-BHC
Aqueous/Solids	EPA 8081A,B	GC/ECD	a-Chlordane
Aqueous/Solids	EPA 8081A,B	GC/ECD	Aldrin
Aqueous/Solids	EPA 8081A,B	GC/ECD	b-BHC
Aqueous/Solids	EPA 8081A,B	GC/ECD	Chlordane
Aqueous/Solids	EPA 8081A,B	GC/ECD	d-BHC
Aqueous/Solids	EPA 8081A,B	GC/ECD	Dieldrin
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endosulfan I
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endosulfan II
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endosulfan sulfate
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endrin
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endrin aldehyde
Aqueous/Solids	EPA 8081A,B	GC/ECD	Endrin ketone
Aqueous/Solids	EPA 8081A,B	GC/ECD	g-BHC (Lindane)
Aqueous/Solids	EPA 8081A,B	GC/ECD	g-Chlordane
Aqueous/Solids	EPA 8081A,B	GC/ECD	Heptachlor
Aqueous/Solids	EPA 8081A,B	GC/ECD	Heptachlor epoxide
Aqueous/Solids	EPA 8081A,B	GC/ECD	Hexachlorobenzene
Aqueous/Solids	EPA 8081A,B	GC/ECD	Methoxychlor
Aqueous/Solids	EPA 8081A,B	GC/ECD	Toxaphene
Aqueous/Solids	EPA 8082A	GC/ECD	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)
Aqueous/Solids	EPA 8082A	GC/ECD	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)
Aqueous/Solids	EPA 8082A	GC/ECD	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)
Aqueous/Solids	EPA 8082A	GC/ECD	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)
Aqueous/Solids	EPA 8082A	GC/ECD	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)



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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8082A	GC/ECD	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)
Aqueous/Solids	EPA 8082A	GC/ECD	2,3',4,4',5'-Pentachlorobiphenyl (PCB 123)
Aqueous/Solids	EPA 8082A	GC/ECD	2,4,4'-Trichlorobiphenyl (PCB 28)
Aqueous/Solids	EPA 8082A	GC/ECD	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)
Aqueous/Solids	EPA 8082A	GC/ECD	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)
Aqueous/Solids	EPA 8082A	GC/ECD	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)
Aqueous/Solids	EPA 8082A	GC/ECD	3,4,4',5-Tetrachlorobiphenyl (PCB 81)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor 1016/1242
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1016 (PCB-1016)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1221 (PCB-1221)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1232 (PCB-1232)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1242 (PCB-1242)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1248 (PCB-1248)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1254 (PCB-1254)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1260 (PCB-1260)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1262 (PCB-1262)
Aqueous/Solids	EPA 8082A	GC/ECD	Aroclor-1268 (PCB-1268)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (129)+(138)+(163)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (153)+(168)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (156)+(157)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (180)+(193)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (20)+(28)
Aqueous/Solids	EPA 8082A	GC/ECD	PCB (90)+(101)+(113)
Aqueous/Solids	EPA 8082A	GC/ECD	PCBs, total
Aqueous/Solids	EPA 8141A,B	GC/NPD	Ametryn
Aqueous/Solids	EPA 8141A,B	GC/NPD	Atraton
Aqueous/Solids	EPA 8141A,B	GC/NPD	Atrazine
Aqueous/Solids	EPA 8141A,B	GC/NPD	Azinphosmethyl
Aqueous/Solids	EPA 8141A,B	GC/NPD	Bolstar
Aqueous/Solids	EPA 8141A,B	GC/NPD	Chlorpyrifos
Aqueous/Solids	EPA 8141A,B	GC/NPD	Coumaphos
Aqueous/Solids	EPA 8141A,B	GC/NPD	Cyanazine



# Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

## APPL, Inc.

908 N. Temperance Avenue, Clovis, CA 93611  
Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8141A,B	GC/NPD	DEF
Aqueous/Solids	EPA 8141A,B	GC/NPD	Demeton, (Mix of Isomers O:S)
Aqueous/Solids	EPA 8141A,B	GC/NPD	Diazinon
Aqueous/Solids	EPA 8141A,B	GC/NPD	Dichlorvos
Aqueous/Solids	EPA 8141A,B	GC/NPD	Dimethoate
Aqueous/Solids	EPA 8141A,B	GC/NPD	Disulfoton
Aqueous/Solids	EPA 8141A,B	GC/NPD	EPN
Aqueous/Solids	EPA 8141A,B	GC/NPD	Ethion
Aqueous/Solids	EPA 8141A,B	GC/NPD	Ethoprop
Aqueous/Solids	EPA 8141A,B	GC/NPD	Fenclorphos (Ronnel)
Aqueous/Solids	EPA 8141A,B	GC/NPD	Fensulfothion
Aqueous/Solids	EPA 8141A,B	GC/NPD	Fenthion
Aqueous/Solids	EPA 8141A,B	GC/NPD	Malathion
Aqueous/Solids	EPA 8141A,B	GC/NPD	Merphos
Aqueous/Solids	EPA 8141A,B	GC/NPD	Mevinphos
Aqueous/Solids	EPA 8141A,B	GC/NPD	Naled
Aqueous/Solids	EPA 8141A,B	GC/NPD	Parathion ethyl
Aqueous/Solids	EPA 8141A,B	GC/NPD	Parathion methyl
Aqueous/Solids	EPA 8141A,B	GC/NPD	Phorate
Aqueous/Solids	EPA 8141A,B	GC/NPD	Prometon
Aqueous/Solids	EPA 8141A,B	GC/NPD	Prometryn
Aqueous/Solids	EPA 8141A,B	GC/NPD	Propazine
Aqueous/Solids	EPA 8141A,B	GC/NPD	Prowl
Aqueous/Solids	EPA 8141A,B	GC/NPD	Simazine
Aqueous/Solids	EPA 8141A,B	GC/NPD	Simetryn
Aqueous/Solids	EPA 8141A,B	GC/NPD	Sulfotep
Aqueous/Solids	EPA 8141A,B	GC/NPD	Terbutryn
Aqueous/Solids	EPA 8141A,B	GC/NPD	Terbutylazine
Aqueous/Solids	EPA 8141A,B	GC/NPD	Tetrachlorvinphos (Stirophos)
Aqueous/Solids	EPA 8141A,B	GC/NPD	Tokuthion
Aqueous/Solids	EPA 8141A,B	GC/NPD	Trichlorinate
Aqueous/Solids	EPA 8141A,B	GC/NPD	Trifluralin
Aqueous/Solids	EPA 8151A	GC/ECD	2,4,5-T
Aqueous/Solids	EPA 8151A	GC/ECD	2,4-D (2,4-Dichlorophenoxyacetic acid)
Aqueous/Solids	EPA 8151A	GC/ECD	2,4-DB



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Diane Anderson Phone: 559-275-2175

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8151A	GC/ECD	3,5-Dichlorobenzoic acid
Aqueous/Solids	EPA 8151A	GC/ECD	4-Nitrophenol
Aqueous/Solids	EPA 8151A	GC/ECD	Acifluorfen
Aqueous/Solids	EPA 8151A	GC/ECD	Bentazon
Aqueous/Solids	EPA 8151A	GC/ECD	Dacthal
Aqueous/Solids	EPA 8151A	GC/ECD	Dalapon
Aqueous/Solids	EPA 8151A	GC/ECD	Dicamba
Aqueous/Solids	EPA 8151A	GC/ECD	Dichlorprop
Aqueous/Solids	EPA 8151A	GC/ECD	Dinoseb (2-sec-Butyl-4,6-dinitrophenol)
Aqueous/Solids	EPA 8151A	GC/ECD	Pentachlorophenol
Aqueous/Solids	EPA 8151A	GC/ECD	Picloram
Aqueous/Solids	EPA 8151A	GC/ECD	Silvex (2,4,5-TP)
Aqueous/Solids	EPA 8260B, C	GC/MS	Cyclohexane
Aqueous/Solids	EPA 8260B, C	GC/MS	Methylacetate
Aqueous/Solids	EPA 8260B, C	GC/MS	Methylcyclohexane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1,1,2-Tetrachloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1,1-Trichloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1,2,2-Tetrachloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1,2-Trichloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1,2-Trichlorotrifluoroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1-Dichloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1-Dichloroethene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,1-Dichloropropene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2,3-Trichlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2,3-Trichloropropane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2,4-Trichlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2,4-Trimethylbenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2-Dibromo-3-chloropropane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2-Dibromoethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2-Dichlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2-Dichloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,2-Dichloropropane
Aqueous/Solids	EPA 8260B,C	GC/MS	1,3,5-Trimethylbenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,3-Dichlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	1,3-Dichloropropane



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*Accreditation is granted to the facility to perform the following testing:*

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8260B,C	GC/MS	1,4-Dichlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	2,2-Dichloropropane
Aqueous/Solids	EPA 8260B,C	GC/MS	2-Butanone (Methyl ethyl ketone)
Aqueous/Solids	EPA 8260B,C	GC/MS	2-Chloroethyl vinyl ether
Aqueous/Solids	EPA 8260B,C	GC/MS	2-Chlorotoluene
Aqueous/Solids	EPA 8260B,C	GC/MS	2-Hexanone
Aqueous/Solids	EPA 8260B,C	GC/MS	4-Chlorotoluene
Aqueous/Solids	EPA 8260B,C	GC/MS	4-methyl-2-pentanone
Aqueous/Solids	EPA 8260B,C	GC/MS	Acetone
Aqueous/Solids	EPA 8260B,C	GC/MS	Acetonitrile
Aqueous/Solids	EPA 8260B,C	GC/MS	Acrolein
Aqueous/Solids	EPA 8260B,C	GC/MS	Acrylonitrile
Aqueous/Solids	EPA 8260B,C	GC/MS	Benzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Bromobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Bromochloromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Bromodichloromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Bromoform
Aqueous/Solids	EPA 8260B,C	GC/MS	Bromomethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Carbon disulphide
Aqueous/Solids	EPA 8260B,C	GC/MS	Carbon tetrachloride
Aqueous/Solids	EPA 8260B,C	GC/MS	Chlorobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Chloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Chloroform
Aqueous/Solids	EPA 8260B,C	GC/MS	Chloromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	cis-1,2-Dichloroethene
Aqueous/Solids	EPA 8260B,C	GC/MS	cis-1,3-Dichloropropene
Aqueous/Solids	EPA 8260B,C	GC/MS	Dibromochloromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Dibromomethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Dichlorodifluoromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Ethyl tert-butyl ether (ETBE)
Aqueous/Solids	EPA 8260B,C	GC/MS	Ethylbenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Hexachlorobutadiene
Aqueous/Solids	EPA 8260B,C	GC/MS	Hexachloroethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Iodomethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Isopropyl ether (DIPE)



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## APPL, Inc.

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Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8260B,C	GC/MS	Isopropylbenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	m+p-Xylene
Aqueous/Solids	EPA 8260B,C	GC/MS	Methyl tert-butyl ether (MTBE)
Aqueous/Solids	EPA 8260B,C	GC/MS	Methylene chloride (Dichloromethane)
Aqueous/Solids	EPA 8260B,C	GC/MS	Naphthalene
Aqueous/Solids	EPA 8260B,C	GC/MS	n-Butyl benzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Nitrobenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	n-Propylbenzene
Aqueous/Solids	EPA 8260B,C	GC/MS	o-Xylene
Aqueous/Solids	EPA 8260B,C	GC/MS	p-isopropyl toluene
Aqueous/Solids	EPA 8260B,C	GC/MS	sec-Butyl benzene
Aqueous/Solids	EPA 8260B,C	GC/MS	Styrene
Aqueous/Solids	EPA 8260B,C	GC/MS	tert-Amyl methyl ether (TAME)
Aqueous/Solids	EPA 8260B,C	GC/MS	tert-Butyl alcohol (t-Butanol)
Aqueous/Solids	EPA 8260B,C	GC/MS	tert-Butyl benzene
Aqueous/Solids	EPA 8260B,C	GC/MS	tert-Butyl ethyl ether (ETBE)
Aqueous/Solids	EPA 8260B,C	GC/MS	Tetrachloroethene
Aqueous/Solids	EPA 8260B,C	GC/MS	Toluene
Aqueous/Solids	EPA 8260B,C	GC/MS	Total Xylenes
Aqueous/Solids	EPA 8260B,C	GC/MS	trans-1,2-Dichloroethene
Aqueous/Solids	EPA 8260B,C	GC/MS	trans-1,3-Dichloropropene
Aqueous/Solids	EPA 8260B,C	GC/MS	Trichloroethene
Aqueous/Solids	EPA 8260B,C	GC/MS	Trichlorofluoromethane
Aqueous/Solids	EPA 8260B,C	GC/MS	Vinyl Acetate
Aqueous/Solids	EPA 8260B,C	GC/MS	Vinyl chloride
Aqueous/Solids	EPA 8270C,D	GC/MS	1,1-Biphenyl
Aqueous/Solids	EPA 8270C,D	GC/MS	1,2,4,5-Tetrachlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	1,2,4-Trichlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	1,2-Dichlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	1,3-Dichlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	1,4-Dichlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	1,4-Dioxane
Aqueous/Solids	EPA 8270C,D	GC/MS	2,3,4,6-Tetrachlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4,5-Trichlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4,6-Trichlorophenol



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## APPL, Inc.

908 N. Temperance Avenue, Clovis, CA 93611  
Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4-Dichlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4-Dimethylphenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4-Dinitrophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,4-Dinitrotoluene (2,4-DNT)
Aqueous/Solids	EPA 8270C,D	GC/MS	2,6-Dichlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2,6-Dinitrotoluene (2,6-DNT)
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Chloronaphthalene
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Chlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Methyl-4,6-Dinitrophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Methylnaphthalene
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Methylphenol (o-Cresol)
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Nitroaniline
Aqueous/Solids	EPA 8270C,D	GC/MS	2-Nitrophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	3,3'-Dichlorobenzidine
Aqueous/Solids	EPA 8270C,D	GC/MS	3+4-Methylphenol (m+p-Cresol)
Aqueous/Solids	EPA 8270C,D	GC/MS	3-Nitroaniline
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Bromophenyl phenyl ether
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Chloro-3-methylphenol
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Chloroaniline
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Chlorophenyl phenylether
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Methylphenol (p-Cresol)
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Nitroaniline
Aqueous/Solids	EPA 8270C,D	GC/MS	4-Nitrophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	Acenaphthene
Aqueous/Solids	EPA 8270C,D	GC/MS	Acenaphthylene
Aqueous/Solids	EPA 8270C,D	GC/MS	Acetophenone
Aqueous/Solids	EPA 8270C,D	GC/MS	Aniline
Aqueous/Solids	EPA 8270C,D	GC/MS	Anthracene
Aqueous/Solids	EPA 8270C,D	GC/MS	Atrazine
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzaldehyde
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzidine
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzo(a)anthracene
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzo(a)pyrene
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzo(b)fluoranthene
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzo(g,h,i)perylene



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<b>Matrix</b>	<b>Standard/Method</b>	<b>Technology</b>	<b>Analyte</b>
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzo(k)fluoranthene
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzoic acid
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzyl alcohol
Aqueous/Solids	EPA 8270C,D	GC/MS	Benzyl butyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Biphenyl
Aqueous/Solids	EPA 8270C,D	GC/MS	bis(2-Chloroethoxy) methane
Aqueous/Solids	EPA 8270C,D	GC/MS	bis(2-Chloroethyl) ether
Aqueous/Solids	EPA 8270C,D	GC/MS	bis(2-Chloroisopropyl) ether
Aqueous/Solids	EPA 8270C,D	GC/MS	bis(2-Ethylhexyl) phthalate (DEHP)
Aqueous/Solids	EPA 8270C,D	GC/MS	Butyl benzyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Caprolactam
Aqueous/Solids	EPA 8270C,D	GC/MS	Carbazole
Aqueous/Solids	EPA 8270C,D	GC/MS	Chrysene
Aqueous/Solids	EPA 8270C,D	GC/MS	Dibenz(a,h) anthracene
Aqueous/Solids	EPA 8270C,D	GC/MS	Dibenzofuran
Aqueous/Solids	EPA 8270C,D	GC/MS	Diethyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Dimethyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Di-n-butyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Di-n-octyl phthalate
Aqueous/Solids	EPA 8270C,D	GC/MS	Fluoranthene
Aqueous/Solids	EPA 8270C,D	GC/MS	Fluorene
Aqueous/Solids	EPA 8270C,D	GC/MS	Hexachlorobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	Hexachlorobutadiene
Aqueous/Solids	EPA 8270C,D	GC/MS	Hexachlorocyclopentadiene
Aqueous/Solids	EPA 8270C,D	GC/MS	Hexachloroethane
Aqueous/Solids	EPA 8270C,D	GC/MS	Indeno(1,2,3-cd) pyrene
Aqueous/Solids	EPA 8270C,D	GC/MS	Isophorone
Aqueous/Solids	EPA 8270C,D	GC/MS	Naphthalene
Aqueous/Solids	EPA 8270C,D	GC/MS	Nitrobenzene
Aqueous/Solids	EPA 8270C,D	GC/MS	N-nitrosodimethylamine
Aqueous/Solids	EPA 8270C,D	GC/MS	N-nitrosodi-n-propylamine
Aqueous/Solids	EPA 8270C,D	GC/MS	n-Nitrosodiphenylamine
Aqueous/Solids	EPA 8270C,D	GC/MS	Pentachlorophenol
Aqueous/Solids	EPA 8270C,D	GC/MS	Phenanthrene
Aqueous/Solids	EPA 8270C,D	GC/MS	Phenol



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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solids	EPA 8270C,D	GC/MS	Pyrene
Aqueous/Solids	EPA 8270C,D	GC/MS	Pyridine
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	1-Methylnaphthalene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	2-Methylnaphthalene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Acenaphthene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Acenaphthylene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Anthracene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(a)anthracene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(a)pyrene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(b)fluoranthene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(b+k)fluoranthene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(e)pyrene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(g,h,i)perylene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Benzo(k)fluoranthene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Chrysene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Dibenzo(a,h)anthracene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Fluoranthene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Fluorene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Indeno(1,2,3-cd) pyrene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Naphthalene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Phenanthrene
Aqueous/Solids	EPA 8270C,D SIM	GC/MS	Pyrene
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,6,7,8,9-OCDD
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,6,7,8,9-OCDF
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,6,7,8-Hpcdd
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,6,7,8-Hpcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,7,8,9-Hpcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,7,8-Hxcdd
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,4,7,8-Hxcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,6,7,8-Hxcdd
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,6,7,8-Hxcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,7,8,9-Hxcdd
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,7,8,9-Hxcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,7,8-Pecdd
Aqueous/Solids	EPA 8290A	HRGC/HRMS	1,2,3,7,8-Pecdf



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Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

<b>Matrix</b>	<b>Standard/Method</b>	<b>Technology</b>	<b>Analyte</b>
Aqueous/Solids	EPA 8290A	HRGC/HRMS	2,3,4,6,7,8-Hxcdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	2,3,4,7,8-Pecdf
Aqueous/Solids	EPA 8290A	HRGC/HRMS	2,3,7,8-TCDD
Aqueous/Solids	EPA 8290A	HRGC/HRMS	2,3,7,8-TCDF
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Hpcdd, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Hpcdf, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Hxcd, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Hxcdf, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	PCDD + PCDF, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	PCDD, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	PCDF, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Pecdd, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	Pecdf, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	TCDD, total
Aqueous/Solids	EPA 8290A	HRGC/HRMS	TCDF, total
Aqueous/Solids	EPA 8321A	HPLC	3-Hydroxycarbofuran
Aqueous/Solids	EPA 8321A	HPLC	Aldicarb
Aqueous/Solids	EPA 8321A	HPLC	Aldicarb sulfone
Aqueous/Solids	EPA 8321A	HPLC	Aldicarb sulfoxide
Aqueous/Solids	EPA 8321A	HPLC	Ammonium picrate
Aqueous/Solids	EPA 8321A	HPLC	Barban
Aqueous/Solids	EPA 8321A	HPLC	Baygon (Propoxur)
Aqueous/Solids	EPA 8321A	HPLC	Bromacil
Aqueous/Solids	EPA 8321A	HPLC	Carbaryl
Aqueous/Solids	EPA 8321A	HPLC	Carbofuran
Aqueous/Solids	EPA 8321A	HPLC	Chloroxuron
Aqueous/Solids	EPA 8321A	HPLC	Dioxacarb
Aqueous/Solids	EPA 8321A	HPLC	Diuron
Aqueous/Solids	EPA 8321A	HPLC	Linuron
Aqueous/Solids	EPA 8321A	HPLC	Methiocarb
Aqueous/Solids	EPA 8321A	HPLC	Methomyl
Aqueous/Solids	EPA 8321A	HPLC	Oxamyl
Aqueous/Solids	EPA 8321A	HPLC	Picric Acid
Aqueous/Solids	EPA 8321A	HPLC	Promecarb
Aqueous/Solids	EPA 8321A	HPLC	Propham



*Certificate of Accreditation: Supplement*  
ISO/IEC 17025:2005 and DoD-ELAP

**APPL, Inc.**

908 N. Temperance Avenue, Clovis, CA 93611  
Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

<b>Matrix</b>	<b>Standard/Method</b>	<b>Technology</b>	<b>Analyte</b>
Aqueous/Solids	EPA 8330A,B	HPLC	1,3,5-Trinitrobenzene
Aqueous/Solids	EPA 8330A,B	HPLC	1,3-Dinitrobenzene
Aqueous/Solids	EPA 8330A,B	HPLC	2,4,6-Trinitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	2,4-Dinitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	2,6-Dinitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	2-Amino-4,6-dinitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	2-Nitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	3-Nitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	4-Amino-2,6-dinitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	4-Nitrotoluene
Aqueous/Solids	EPA 8330A,B	HPLC	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Aqueous/Solids	EPA 8330A,B	HPLC	Nitrobenzene
Aqueous/Solids	EPA 8330A,B	HPLC	Nitroglycerin
Aqueous/Solids	EPA 8330A,B	HPLC	Pentaerythritoltetranitrate (PETN)
Aqueous/Solids	EPA 8330A,B	HPLC	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Aqueous/Solids	EPA 8330A,B	HPLC	Tetryl (Methyl-2,4,6-trinitrophenylnitramine)
Aqueous/Solids	EPA 9010C	Distillation/UV/Vis	Amenable Cyanide
Aqueous/Solids	EPA 9010C	Distillation/UV/Vis	Total Cyanide
Aqueous/Solids	EPA 9010C	UV/Vis	Total Cyanide
Aqueous/Solids	EPA 9014	Distillation/UV/Vis	Amenable Cyanide
Aqueous/Solids	EPA 9014	Distillation/UV/Vis	Total Cyanide
Aqueous/Solids	EPA 9014	UV/Vis	Total Cyanide
Aqueous/Solids	EPA 9040C	Ion Selective Electrode	pH/Corrosivity
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Bromide
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Chloride
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Fluoride
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Nitrate as N
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Nitrite + Nitrate as N
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Nitrite as N
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Orthophosphate as P
Aqueous/Solids	EPA 9056A	Ion Chromatography (IC)	Sulfate



# Certificate of Accreditation: Supplement

## ISO/IEC 17025:2005 and DoD-ELAP

### APPL, Inc.

908 N. Temperance Avenue, Clovis, CA 93611  
Diane Anderson Phone: 559-275-2175

*Accreditation is granted to the facility to perform the following testing:*

<b>Matrix</b>	<b>Standard/Method</b>	<b>Technology</b>	<b>Analyte</b>
Aqueous	EPA 3010A	Hot Block	Acid digestion for metals analysis
Aqueous	EPA 3015A	Microwave	Microwave assisted acid digestion for metals analysis
Aqueous	EPA 3510C	Separatory funnel	Separatory funnel extraction
Aqueous	EPA 3520C	Liquid-liquid extractor	Liquid-Liquid extraction
Aqueous	EPA 3535A	SPE	SPE extraction for explosives
Aqueous	EPA 5030B,C	Purge and trap	Purge and trap
Aqueous	EPA 7470A	Hotplate digestion	Mercury digestion
Solids	CCR Chapter 11, Article 5, Appendix II	Rotary tumbler	Waste Extraction test (WET) (STLC)
Solids	EPA 1311	Rotary tumbler	TCLP Extraction
Solids	EPA 1312	Rotary tumbler	SPLP Extraction
Solids	EPA 3050B	Hotplate digestion	Acid digestion for metals analysis
Solids	EPA 3051A	Microwave	Microwave assisted acid digestion for metals analysis
Solids	EPA 3060A	Hotplate digestion	Alkaline digestion for hexavalent chromium
Solids	EPA 3550B	Ultrasonic waterbath	Ultrasonic extraction
Solids	EPA 5035A	Closed-system purge and trap	Closed-system purge and trap extraction
Solids	EPA 7471B	Hotplate digestion	Mercury digestion
Solids	EPA 8330B, Appendix A	Puck mill grinder	Incremental sampling
Aqueous/Solids	EPA 3540C	Soxhlet	Soxhlet extraction
Aqueous/Solids	EPA 3630C	Cleanup	Silica gel cleanup
Aqueous/Solids	EPA 3660B	Cleanup	Sulfuric acid cleanup
Aqueous/Solids	EPA 3665A	Cleanup	Sulfuric acid – Permanganate cleanup
Aqueous/Solids	EPA 8151A	Separatory funnel	Herbicide extraction

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CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

**CERTIFICATE OF ENVIRONMENTAL LABORATORY ACCREDITATION**

Is hereby granted to

**APPL, Inc. (Agriculture & Priority Pollutants Laboratories, Inc.)**

908 North Temperance Avenue

Clovis, CA 93611

Scope of the certificate is limited to the  
"Fields of Testing"  
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site,  
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of  
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **1312**

Expiration Date: **09/30/2016**

Effective Date: **10/01/2014**

Richmond, California  
subject to forfeiture or revocation

A handwritten signature in black ink, appearing to read "Christine Sotelo".

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Christine Sotelo, Chief  
California State Environmental Laboratory Accreditation Program

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**LABORATORY  
ACCREDITATION  
BUREAU** a division of A-5-B



# Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2278

**EMAX Laboratories, Inc.**

1835 W. 205<sup>th</sup> St.  
Torrance CA 90501

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).\*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: January 10, 2017

**R. Douglas Leonard, Jr., President, COO**  
**Laboratory Accreditation Bureau**  
**Presented the 9<sup>th</sup> of January 2014**

\*See the laboratory's Scope of Accreditation for details of accredited parameters

\*\*Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).  
Form 403.14 - Rev 1 7/3/13

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## Scope of Accreditation For EMAX Laboratories, Inc.

1835 W 205<sup>th</sup> Street  
Torrance, CA 90501  
Kenette Pimentel  
310-618-8889

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **EMAX Laboratories, Inc.** to perform the following tests:

Accreditation granted through: **January 10, 2017**

### Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC	AK101	GRO
GC	AK102	DRO
GFAA	CA 939M	Organo Lead
Platinum Electrode	EPA 120.1	Specific Conductance
Titrimetric	EPA 130.2	Hardness
Electrode	EPA 150.1	pH
Gravimetric	EPA 160.1	TDS
Gravimetric	EPA 160.2	TSS
Gravimetric	EPA 160.3	Total Residue
Turbidimetric	EPA 180.1	Turbidity
ICP	EPA 200.7	Aluminum
ICP	EPA 200.7	Antimony
ICP	EPA 200.7	Arsenic
ICP	EPA 200.7	Barium
ICP	EPA 200.7	Beryllium
ICP	EPA 200.7	Boron
ICP	EPA 200.7	Cadmium
ICP	EPA 200.7	Calcium
ICP	EPA 200.7	Chromium
ICP	EPA 200.7	Cobalt
ICP	EPA 200.7	Copper
ICP	EPA 200.7	Iron



<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
ICP	EPA 200.7	Lead
ICP	EPA 200.7	Magnesium
ICP	EPA 200.7	Manganese
ICP	EPA 200.7	Molybdenum
ICP	EPA 200.7	Nickel
ICP	EPA 200.7	Potassium
ICP	EPA 200.7	Selenium
ICP	EPA 200.7	Silver
ICP	EPA 200.7	Sodium
ICP	EPA 200.7	Strontium
ICP	EPA 200.7	Thallium
ICP	EPA 200.7	Tin
ICP	EPA 200.7	Titanium
ICP	EPA 200.7	Vanadium
ICP	EPA 200.7	Zinc
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Chromium
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
ICP-MS	EPA 200.8	Zinc
IC	EPA 218.6	Hexavalent Chromium
COLD VAPOR	EPA 245.1	Mercury
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Chloride
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Bromide
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Phosphate
IC	EPA 300.0	Sulfate
IC	EPA 300.0	Bromate
IC	EPA 300.0	Chlorate
IC	EPA 300M	Lactate
IC	EPA 300M	Acetate
IC	EPA 300M	Propionate
IC	EPA 300M	Butyrate
IC	EPA 300M	Pyruvate
IC	EPA 310.1	Alkalinity
IC	EPA 314.0	Perchlorate
Titrimetric	EPA 330.3	Total Residual Chlorine
Spectrometric	EPA 352.1	Nitrate-N
Spectrometric	EPA 353.3	Nitrate-N
Spectrometric	EPA 354.1	Nitrite-N
Spectrometric	EPA 365.2	Ortho-phosphate
Spectrometric	EPA 335.2	Cyanide
Spectrometric	EPA 350.2	Ammonia
Spectrometric	EPA 351.3	TKN
Spectrometric	EPA 365.2	Phosphorus
Spectrometric	EPA 370.1	Silica
Titrimetric	EPA 376.1	Sulfide
Spectrometric	EPA 376.2	Sulfide
Electrode	EPA 405.1	BOD
Spectrometric	EPA 410.4	COD
Combustion-IR	EPA 415.1	TOC
Spectrometric	EPA 420.1	Phenols
Spectrometric	EPA 425.1	MBAS
GC	EPA 504.1	DBCP
GC	EPA 504.1	EDB
GC	EPA 608	Aldrin
GC	EPA 608	alpha-BHC
GC	EPA 608	beta-BHC
GC	EPA 608	delta-BHC
GC	EPA 608	gamma-BHC (Lindane)

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 608	DDD (4,4)
GC	EPA 608	DDE (4,4)
GC	EPA 608	DDT (4,4)
GC	EPA 608	Dieldrin
GC	EPA 608	Endosulfan I
GC	EPA 608	Endosulfan II
GC	EPA 608	Endosulfan sulfate
GC	EPA 608	Endrin
GC	EPA 608	Endrin Aldehyde
GC	EPA 608	Heptachlor
GC	EPA 608	Heptachlor epoxide
GC	EPA 608	Methoxychlor
GC	EPA 608	alpha-Chlordane
GC	EPA 608	gamma-Chlordane
GC	EPA 608	Endrin Ketone
GC	EPA 608	Toxaphene
GC	EPA 608	Technical Chlordane
GC	EPA 608	cis-Nonachlor
GC	EPA 608	DDD (2,4)
GC	EPA 608	DDE (2,4)
GC	EPA 608	DDT (2,4)
GC	EPA 608	Mirex
GC	EPA 608	Oxychlordane
GC	EPA 608	trans-Nonachlor
GC	EPA 608	PCB1016
GC	EPA 608	PCB1221
GC	EPA 608	PCB1232
GC	EPA 608	PCB1242
GC	EPA 608	PCB1248
GC	EPA 608	PCB1254
GC	EPA 608	PCB1260
GC	EPA 608	PCB1262
GC	EPA 608	PCB1268
GC-MS	EPA 624	Acrolein
GC-MS	EPA 624	Acrylonitrile
GC-MS	EPA 624	Benzene
GC-MS	EPA 624	Bromodichloromethane
GC-MS	EPA 624	Bromoform
GC-MS	EPA 624	Bromomethane
GC-MS	EPA 624	Carbon tetrachloride
GC-MS	EPA 624	Chlorobenzene
GC-MS	EPA 624	2-Chloroethyl vinyl ether
GC-MS	EPA 624	Chloroethane

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 624	Chloroform
GC-MS	EPA 624	Chloromethane
GC-MS	EPA 624	Dibromochloromethane
GC-MS	EPA 624	1,1-Dichloroethane
GC-MS	EPA 624	1,2-Dichloroethane
GC-MS	EPA 624	1,2-Dichlorobenzene
GC-MS	EPA 624	1,3-Dichlorobenzene
GC-MS	EPA 624	1,4-Dichlorobenzene
GC-MS	EPA 624	Dichlorodifluoromethane
GC-MS	EPA 624	1,1-Dichloroethene
GC-MS	EPA 624	cis-1,2-Dichloroethene
GC-MS	EPA 624	trans-1,2-Dichloroethene
GC-MS	EPA 624	1,2-Dichloropropane
GC-MS	EPA 624	cis-1,3-Dichloropropene
GC-MS	EPA 624	trans-1,3-Dichloropropene
GC-MS	EPA 624	Ethylbenzene
GC-MS	EPA 624	Methylene Chloride
GC-MS	EPA 624	tert-Butyl methyl ether
GC-MS	EPA 624	Styrene
GC-MS	EPA 624	1,1,2,2-Tetrachloroethane
GC-MS	EPA 624	Tetrachloroethene
GC-MS	EPA 624	Toluene
GC-MS	EPA 624	1,1,1-Trichloroethane
GC-MS	EPA 624	1,1,2-Trichloroethane
GC-MS	EPA 624	1,2,4-Trichlorobenzene
GC-MS	EPA 624	Trichloroethene
GC-MS	EPA 624	Trichlorofluoromethane
GC-MS	EPA 624	1,1,2-Trichloro 1,2,2-trifluoroethane
GC-MS	EPA 624	Vinyl Chloride
GC-MS	EPA 624	m-Xylene & p-xylene
GC-MS	EPA 624	o-Xylene
GC-MS	EPA 625	Acenaphthene
GC-MS	EPA 625	Acenaphthylene
GC-MS	EPA 625	Aniline
GC-MS	EPA 625	Anthracene
GC-MS	EPA 625	Azobenzene
GC-MS	EPA 625	Benzidine
GC-MS	EPA 625	Benzo(a)anthracene
GC-MS	EPA 625	benzo(a)pyrene
GC-MS	EPA 625	Benzo(b)fluoranthene
GC-MS	EPA 625	Benzo(e)pyrene
GC-MS	EPA 625	Benzo(g,h,i)perylene
GC-MS	EPA 625	Benzo(k)fluoranthene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 625	Benzoic Acid
GC-MS	EPA 625	Benzyl Alcohol
GC-MS	EPA 625	Biphenyl
GC-MS	EPA 625	bis(2-chloroethoxy)methane
GC-MS	EPA 625	bis(2-chloroethyl)ether
GC-MS	EPA 625	bis(2-chloroisopropyl)ether
GC-MS	EPA 625	bis(2-Ethylhexyl)adipate
GC-MS	EPA 625	bis(2-Ethylhexyl)phthalate
GC-MS	EPA 625	4-Bromophenyl-phenylether
GC-MS	EPA 625	Butylbenzylphthalate
GC-MS	EPA 625	Carbazole
GC-MS	EPA 625	4-Chloro-3-methylphenol
GC-MS	EPA 625	4-Chloroaniline
GC-MS	EPA 625	2-Chloronaphthalene
GC-MS	EPA 625	2-Chlorophenol
GC-MS	EPA 625	4-Chlorophenyl-phenylether
GC-MS	EPA 625	Chrysene
GC-MS	EPA 625	Dibenzo(a,h)anthracene
GC-MS	EPA 625	Dibenzofuran
GC-MS	EPA 625	1,2-Dichlorobenzene
GC-MS	EPA 625	1,3-Dichlorobenzene
GC-MS	EPA 625	1,4-Dichlorobenzene
GC-MS	EPA 625	3,3'-Dichlorobenzidine
GC-MS	EPA 625	2,4-Dichlorophenol
GC-MS	EPA 625	Diethylphthalate
GC-MS	EPA 625	2,6-Dimethylnaphthalene
GC-MS	EPA 625	2,4-Dimethylphenol
GC-MS	EPA 625	Dimethylphthalate
GC-MS	EPA 625	Di-n-butylphthalate
GC-MS	EPA 625	4,6-Dinitro-2-methylphenol
GC-MS	EPA 625	2,4-Dinitrophenol
GC-MS	EPA 625	2,4-Dinitrotoluene
GC-MS	EPA 625	2,6-Dinitrotoluene
GC-MS	EPA 625	Di-n-octylphthalate
GC-MS	EPA 625	1,2-Diphenylhydrazine
GC-MS	EPA 625	Fluoranthene
GC-MS	EPA 625	Fluorene
GC-MS	EPA 625	Hexachlorobenzene
GC-MS	EPA 625	Hexachlorobutadiene
GC-MS	EPA 625	Hexachlorocyclopentadiene
GC-MS	EPA 625	Hexachloroethane
GC-MS	EPA 625	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 625	Isophorone

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 625	1-Methylnaphthalene
GC-MS	EPA 625	2-Methylnaphthalene
GC-MS	EPA 625	1-Methylphenanthrene
GC-MS	EPA 625	2-Methylphenol
GC-MS	EPA 625	4-Methylphenol
GC-MS	EPA 625	Naphthalene
GC-MS	EPA 625	2-Nitroaniline
GC-MS	EPA 625	3-Nitroaniline
GC-MS	EPA 625	4-Nitroaniline
GC-MS	EPA 625	Nitrobenzene
GC-MS	EPA 625	2-Nitrophenol
GC-MS	EPA 625	4-Nitrophenol
GC-MS	EPA 625	n-Nitrosodimethylamine
GC-MS	EPA 625	n-Nitroso-di-n-propylamine
GC-MS	EPA 625	n-Nitrosodiphenylamine
GC-MS	EPA 625	Pentachlorophenol
GC-MS	EPA 625	Perylene
GC-MS	EPA 625	Phenanthrene
GC-MS	EPA 625	Phenol
GC-MS	EPA 625	Pyrene
GC-MS	EPA 625	Pyridine
GC-MS	EPA 625	2,3,4,6-Tetrachlorophenol
GC-MS	EPA 625	1,2,4-Trichlorobenzene
GC-MS	EPA 625	2,3,4-Trichlorophenol
GC-MS	EPA 625	2,3,5-Trichlorophenol
GC-MS	EPA 625	2,4,5-Trichlorophenol
GC-MS	EPA 625	2,4,6-Trichlorophenol
GC-MS	EPA 625	2,3,5-Trimethylnaphthalene
Gravimetric	EPA 1664A / 1664 B	Oil & Grease
Pensky-Martens	EPA 1010 / 1010A	Ignitability
ICP	EPA 6010B / 6010C	Aluminum
ICP	EPA 6010B / 6010C	Antimony
ICP	EPA 6010B / 6010C	Arsenic
ICP	EPA 6010B / 6010C	Barium
ICP	EPA 6010B / 6010C	Beryllium
ICP	EPA 6010B / 6010C	Boron
ICP	EPA 6010B / 6010C	Cadmium
ICP	EPA 6010B / 6010C	Calcium
ICP	EPA 6010B / 6010C	Chromium
ICP	EPA 6010B / 6010C	Cobalt
ICP	EPA 6010B / 6010C	Copper
ICP	EPA 6010B / 6010C	Iron
ICP	EPA 6010B / 6010C	Lead



<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
ICP	EPA 6010B / 6010C	Magnesium
ICP	EPA 6010B / 6010C	Manganese
ICP	EPA 6010B / 6010C	Molybdenum
ICP	EPA 6010B / 6010C	Nickel
ICP	EPA 6010B / 6010C	Potassium
ICP	EPA 6010B / 6010C	Selenium
ICP	EPA 6010B / 6010C	Silver
ICP	EPA 6010B / 6010C	Sodium
ICP	EPA 6010B / 6010C	Strontium
ICP	EPA 6010B / 6010C	Thallium
ICP	EPA 6010B / 6010C	Tin
ICP	EPA 6010B / 6010C	Titanium
ICP	EPA 6010B / 6010C	Vanadium
ICP	EPA 6010B / 6010C	Zinc
ICP-MS	EPA 6020A	Aluminum
ICP-MS	EPA 6020A	Antimony
ICP-MS	EPA 6020A	Arsenic
ICP-MS	EPA 6020A	Barium
ICP-MS	EPA 6020A	Beryllium
ICP-MS	EPA 6020A	Boron
ICP-MS	EPA 6020A	Cadmium
ICP-MS	EPA 6020A	Calcium
ICP-MS	EPA 6020A	Chromium
ICP-MS	EPA 6020A	Cobalt
ICP-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Nickel
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Zinc

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC-MS	EPA 6850	Perchlorate
Spectrometric	EPA 7196A	Hex. Chromium
IC	EPA 7199	Hex. Chromium
Cold-Vapor	EPA 7470A	Mercury
GC	EPA 8015B / 8015C / 8015D	Gasoline
GC	EPA 8015B / 8015C / 8015D	Diesel
GC	EPA 8015B / 8015C / 8015D	Motor Oil
GC	EPA 8015B / 8015C	Diethylene Glycol
GC	EPA 8015B / 8015C	Ethanol
GC	EPA 8015B / 8015C	Ethylene Glycol
GC	EPA 8015B / 8015C	Isopropanol
GC	EPA 8015B / 8015C / 8015D	JP4
GC	EPA 8015B / 8015C	Methanol
GC	EPA 8015B / 8015C	Propylene Glycol
GC	EPA 8015B / 8015C / 8015D	JP5
GC	EPA 8015B / 8015C	Triethylene Glycol
GC	EPA 8081A / 8081B	Aldrin
GC	EPA 8081A / 8081B	alpha-BHC
GC	EPA 8081A / 8081B	beta-BHC
GC	EPA 8081A / 8081B	delta-BHC
GC	EPA 8081A / 8081B	gamma-BHC (Lindane)
GC	EPA 8081A / 8081B	DDD (4,4)
GC	EPA 8081A / 8081B	DDE (4,4)
GC	EPA 8081A / 8081B	DDT (4,4)
GC	EPA 8081A / 8081B	Dieldrin
GC	EPA 8081A / 8081B	Endosulfan I
GC	EPA 8081A / 8081B	Endosulfan II
GC	EPA 8081A / 8081B	Endosulfan sulfate
GC	EPA 8081A / 8081B	Endrin
GC	EPA 8081A / 8081B	Endrin Aldehyde
GC	EPA 8081A / 8081B	Heptachlor
GC	EPA 8081A / 8081B	Heptachlor epoxide
GC	EPA 8081A / 8081B	Methoxychlor
GC	EPA 8081A / 8081B	alpha-Chlordane
GC	EPA 8081A / 8081B	gamma-Chlordane
GC	EPA 8081A / 8081B	Endrin Ketone
GC	EPA 8081A / 8081B	Toxaphene
GC	EPA 8081A / 8081B	Technical Chlordane
GC	EPA 8081A / 8081B	cis-Nonachlor
GC	EPA 8081A / 8081B	DDD (2,4)
GC	EPA 8081A / 8081B	DDE (2,4)
GC	EPA 8081A / 8081B	DDT (2,4)
GC	EPA 8081A / 8081B	Mirex

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8081A / 8081B	Oxychlorthane
GC	EPA 8081A / 8081B	trans-Nonachlor
GC	EPA 8082 / 8082A	PCB1016
GC	EPA 8082 / 8082A	PCB1221
GC	EPA 8082 / 8082A	PCB1232
GC	EPA 8082 / 8082A	PCB1242
GC	EPA 8082 / 8082A	PCB1248
GC	EPA 8082 / 8082A	PCB1254
GC	EPA 8082 / 8082A	PCB1260
GC	EPA 8082 / 8082A	PCB1262
GC	EPA 8082 / 8082A	PCB1268
GC	EPA 8082 / 8082A	PCB 8
GC	EPA 8082 / 8082A	PCB 18
GC	EPA 8082 / 8082A	PCB 28
GC	EPA 8082 / 8082A	PCB 44
GC	EPA 8082 / 8082A	PCB 52
GC	EPA 8082 / 8082A	PCB 66
GC	EPA 8082 / 8082A	PCB 77
GC	EPA 8082 / 8082A	PCB 81
GC	EPA 8082 / 8082A	PCB 101
GC	EPA 8082 / 8082A	PCB 105
GC	EPA 8082 / 8082A	PCB 114
GC	EPA 8082 / 8082A	PCB 118
GC	EPA 8082 / 8082A	PCB 123
GC	EPA 8082 / 8082A	PCB 126
GC	EPA 8082 / 8082A	PCB 128
GC	EPA 8082 / 8082A	PCB 138
GC	EPA 8082 / 8082A	PCB 153
GC	EPA 8082 / 8082A	PCB 156
GC	EPA 8082 / 8082A	PCB 157
GC	EPA 8082 / 8082A	PCB 167
GC	EPA 8082 / 8082A	PCB 169
GC	EPA 8082 / 8082A	PCB 170
GC	EPA 8082 / 8082A	PCB 180
GC	EPA 8082 / 8082A	PCB 187
GC	EPA 8082 / 8082A	PCB 189
GC	EPA 8082 / 8082A	PCB 195
GC	EPA 8082 / 8082A	PCB 206
GC	EPA 8082 / 8082A	PCB 209
GC	EPA 8082 / 8082A	PCB 110
GC	EPA 8141A / 8141B	Azinphos-methyl
GC	EPA 8141A / 8141B	Bolstar
GC	EPA 8141A / 8141B	Chlorpyrifos

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8141A / 8141B	Coumaphos
GC	EPA 8141A / 8141B	Demeton
GC	EPA 8141A / 8141B	Diazinon
GC	EPA 8141A / 8141B	Dichlorvos
GC	EPA 8141A / 8141B	Disulfoton
GC	EPA 8141A / 8141B	Ethoprop
GC	EPA 8141A / 8141B	Fensulfothion
GC	EPA 8141A / 8141B	Fenthion
GC	EPA 8141A / 8141B	Merphos
GC	EPA 8141A / 8141B	Mevinphos
GC	EPA 8141A / 8141B	Naled
GC	EPA 8141A / 8141B	Methyl Parathion
GC	EPA 8141A / 8141B	Phorate
GC	EPA 8141A / 8141B	Ronnel
GC	EPA 8141A / 8141B	Stirophos
GC	EPA 8141A / 8141B	Tokuthion
GC	EPA 8141A / 8141B	Trichloronate
GC	EPA 8141A / 8141B	Dimethoate
GC	EPA 8141A / 8141B	EPN
GC	EPA 8141A / 8141B	Famphur
GC	EPA 8141A / 8141B	Malathion
GC	EPA 8141A / 8141B	Ethyl Parathion
GC	EPA 8141A / 8141B	O,O,O-Triethylphosphorothioate
GC	EPA 8141A / 8141B	Sulfotepp
GC	EPA 8141A / 8141B	Thionazin
GC	EPA 8141A / 8141B	Tributyl Phosphate
GC	EPA 8151A	Acifluorfen
GC	EPA 8151A	Bentazon
GC	EPA 8151A	Chloramben
GC	EPA 8151A	2,4-D
GC	EPA 8151A	2,4-DB
GC	EPA 8151A	Dacthal
GC	EPA 8151A	Dalapon
GC	EPA 8151A	Dicamba
GC	EPA 8151A	3,5-Dichlorobenzoic acid
GC	EPA 8151A	Dichlorprop
GC	EPA 8151A	Dinoseb
GC	EPA 8151A	MCPA
GC	EPA 8151A	MCPP
GC	EPA 8151A	4-Nitrophenol
GC	EPA 8151A	Pentachlorophenol
GC	EPA 8151A	Picloram
GC	EPA 8151A	Silvex

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8151A	2,4,5-T
GC-MS	EPA 8260B / 8260C	Acetone
GC-MS	EPA 8260B / 8260C	Acrolein
GC-MS	EPA 8260B / 8260C	Acrylonitrile
GC-MS	EPA 8260B / 8260C	Benzene
GC-MS	EPA 8260B / 8260C	Bromobenzene
GC-MS	EPA 8260B / 8260C	Bromochloromethane
GC-MS	EPA 8260B / 8260C	Bromodichloromethane
GC-MS	EPA 8260B / 8260C	Bromoform
GC-MS	EPA 8260B / 8260C	Bromomethane
GC-MS	EPA 8260B / 8260C	tert-Butyl alcohol
GC-MS	EPA 8260B / 8260C	2-Butanone (MEK)
GC-MS	EPA 8260B / 8260C	n-Butylbenzene
GC-MS	EPA 8260B / 8260C	sec-Butylbenzene
GC-MS	EPA 8260B / 8260C	tert-Butylbenzene
GC-MS	EPA 8260B / 8260C	Carbon disulfide
GC-MS	EPA 8260B / 8260C	Carbon tetrachloride
GC-MS	EPA 8260B / 8260C	Chlorobenzene
GC-MS	EPA 8260B / 8260C	2-Chloroethyl vinyl ether
GC-MS	EPA 8260B / 8260C	Chloroethane
GC-MS	EPA 8260B / 8260C	Chloroform
GC-MS	EPA 8260B / 8260C	1-Chlorohexane
GC-MS	EPA 8260B / 8260C	Chloromethane
GC-MS	EPA 8260B / 8260C	2-Chlorotoluene
GC-MS	EPA 8260B / 8260C	4-Chlorotoluene
GC-MS	EPA 8260B / 8260C	Isopropyl ether (DIPE)
GC-MS	EPA 8260B / 8260C	Dibromochloromethane
GC-MS	EPA 8260B / 8260C	1,2-Dibromo-3-chloropropane
GC-MS	EPA 8260B / 8260C	1,2-Dibromoethane
GC-MS	EPA 8260B / 8260C	Dibromomethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloroethane
GC-MS	EPA 8260B / 8260C	1,2-Dichloroethane
GC-MS	EPA 8260B / 8260C	1,2-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	1,3-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	trans-1,4-Dichloro-2-Butene
GC-MS	EPA 8260B / 8260C	1,4-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	Dichlorodifluoromethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloroethene
GC-MS	EPA 8260B / 8260C	cis-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C	trans-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C	Dichlorofluoromethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloropropene
GC-MS	EPA 8260B / 8260C	1,2-Dichloropropane

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8260B / 8260C	1,3-Dichloropropane
GC-MS	EPA 8260B / 8260C	2,2-Dichloropropane
GC-MS	EPA 8260B / 8260C	cis-1,3-Dichloropropene
GC-MS	EPA 8260B / 8260C	trans-1,3-Dichloropropene
GC-MS	EPA 8260B / 8260C	tert-Butyl ethyl ether (ETBE)
GC-MS	EPA 8260B / 8260C	Ethyl Methacrylate
GC-MS	EPA 8260B / 8260C	Ethylbenzene
GC-MS	EPA 8260B / 8260C	2-Hexanone (MBK)
GC-MS	EPA 8260B / 8260C	Hexachlorobutadiene
GC-MS	EPA 8260B / 8260C	Iodomethane
GC-MS	EPA 8260B / 8260C	Isopropylbenzene
GC-MS	EPA 8260B / 8260C	p-Isopropyltoluene
GC-MS	EPA 8260B / 8260C	Methylene Chloride
GC-MS	EPA 8260B / 8260C	4-Methyl-2-pentanone (MIBK)
GC-MS	EPA 8260B / 8260C	tert-Butyl methyl ether
GC-MS	EPA 8260B / 8260C	Naphthalene
GC-MS	EPA 8260B / 8260C	n-Propylbenzene
GC-MS	EPA 8260B / 8260C	Styrene
GC-MS	EPA 8260B / 8260C	tert-Amyl methyl ether (TAME)
GC-MS	EPA 8260B / 8260C	1,1,1,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C	1,1,2,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C	Tetrachloroethene
GC-MS	EPA 8260B / 8260C	Toluene
GC-MS	EPA 8260B / 8260C	1,1,1-Trichloroethane
GC-MS	EPA 8260B / 8260C	1,1,2-Trichloroethane
GC-MS	EPA 8260B / 8260C	1,2,3-Trichlorobenzene
GC-MS	EPA 8260B / 8260C	1,2,4-Trichlorobenzene
GC-MS	EPA 8260B / 8260C	Trichloroethene
GC-MS	EPA 8260B / 8260C	Trichlorofluoromethane
GC-MS	EPA 8260B / 8260C	1,2,3-Trichloropropane
GC-MS	EPA 8260B / 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC-MS	EPA 8260B / 8260C	1,2,4-Trimethylbenzene
GC-MS	EPA 8260B / 8260C	1,3,5-Trimethylbenzene
GC-MS	EPA 8260B / 8260C	Vinyl Acetate
GC-MS	EPA 8260B / 8260C	Vinyl Chloride
GC-MS	EPA 8260B / 8260C	m-Xylene & p-xylene
GC-MS	EPA 8260B / 8260C	o-Xylene
GC-MS	EPA 8260B / 8260C	2-Butanol
GC-MS	EPA 8260B / 8260C	Cyclohexane
GC-MS	EPA 8260B / 8260C	1,4-Dioxane
GC-MS	EPA 8260B / 8260C	2-Chloro-1,1,1-trifluoroethane
GC-MS	EPA 8260B / 8260C	Chlorotrifluoroethylene
GC-MS	EPA 8260B / 8260C	cis-1,4-Dichloro-2-butene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8260B / 8260C	Ethanol
GC-MS	EPA 8260B / 8260C	Ethyl Methacrylate
GC-MS	EPA 8260B / 8260C	Isobutyl Alcohol
GC-MS	EPA 8260B / 8260C	Methacrylonitrile
GC-MS	EPA 8260B / 8260C	Methyl Methacrylate
GC-MS	EPA 8260B / 8260C	Pentachloroethane
GC-MS	EPA 8260B / 8260C	Propionitrile
GC-MS	EPA 8260B / 8260C	Sec-Propyl alcohol
GC-MS	EPA 8260B / 8260C	Tetrahydrofuran
GC-MS	EPA 8260B / 8260C	trans-1,4-Dichloro-2-butene
GC-MS	EPA 8260B / 8260C SIM	Benzene
GC-MS	EPA 8260B / 8260C SIM	Carbon tetrachloride
GC-MS	EPA 8260B / 8260C SIM	Chloroform
GC-MS	EPA 8260B / 8260C SIM	Chloromethane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dibromo-3-chloropropane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dibromoethane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dichloroethane
GC-MS	EPA 8260B / 8260C SIM	1,1-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	cis-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	trans-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	1,1,2,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C SIM	Tetrachloroethene
GC-MS	EPA 8260B / 8260C SIM	1,1,1-Trichloroethane
GC-MS	EPA 8260B / 8260C SIM	1,1,2-Trichloroethane
GC-MS	EPA 8260B / 8260C SIM	Trichloroethene
GC-MS	EPA 8260B / 8260C SIM	1,2,3-Trichloropropane
GC-MS	EPA 8260B / 8260C SIM	Vinyl Chloride
GC-MS	EPA 8260B / 8260C SIM	1,4-Dioxane
GC-MS	EPA 8270C / 8270D	Acenaphthene
GC-MS	EPA 8270C / 8270D	Acenaphthylene
GC-MS	EPA 8270C / 8270D	Aniline
GC-MS	EPA 8270C / 8270D	Anthracene
GC-MS	EPA 8270C / 8270D	Azobenzene
GC-MS	EPA 8270C / 8270D	Benzidine
GC-MS	EPA 8270C / 8270D	Benzo(a)anthracene
GC-MS	EPA 8270C / 8270D	benzo(a)pyrene
GC-MS	EPA 8270C / 8270D	Benzo(b)fluoranthene
GC-MS	EPA 8270C / 8270D	Benzo(e)pyrene
GC-MS	EPA 8270C / 8270D	Benzo(g,h,i)perylene
GC-MS	EPA 8270C / 8270D	Benzo(k)fluoranthene
GC-MS	EPA 8270C / 8270D	Benzoic Acid
GC-MS	EPA 8270C / 8270D	Benzyl Alcohol
GC-MS	EPA 8270C / 8270D	Biphenyl

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	bis(2-chloroethoxy)methane
GC-MS	EPA 8270C / 8270D	bis(2-chloroethyl)ether
GC-MS	EPA 8270C / 8270D	bis(2-chloroisopropyl)ether
GC-MS	EPA 8270C / 8270D	bis(2-Ethylhexyl)adipate
GC-MS	EPA 8270C / 8270D	bis(2-Ethylhexyl)phthalate
GC-MS	EPA 8270C / 8270D	4-Bromophenyl-phenylether
GC-MS	EPA 8270C / 8270D	Butylbenzylphthalate
GC-MS	EPA 8270C / 8270D	Carbazole
GC-MS	EPA 8270C / 8270D	4-Chloro-3-methylphenol
GC-MS	EPA 8270C / 8270D	4-Chloroaniline
GC-MS	EPA 8270C / 8270D	2-Chloronaphthalene
GC-MS	EPA 8270C / 8270D	2-Chlorophenol
GC-MS	EPA 8270C / 8270D	4-Chlorophenyl-phenylether
GC-MS	EPA 8270C / 8270D	Chrysene
GC-MS	EPA 8270C / 8270D	Dibenzo(a,h)anthracene
GC-MS	EPA 8270C / 8270D	Dibenzofuran
GC-MS	EPA 8270C / 8270D	1,2-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	1,3-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	1,4-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	3,3'-Dichlorobenzidine
GC-MS	EPA 8270C / 8270D	2,4-Dichlorophenol
GC-MS	EPA 8270C / 8270D	Diethylphthalate
GC-MS	EPA 8270C / 8270D	2,6-Dimethylnaphthalene
GC-MS	EPA 8270C / 8270D	2,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D	Dimethylphthalate
GC-MS	EPA 8270C / 8270D	Di-n-butylphthalate
GC-MS	EPA 8270C / 8270D	4,6-Dinitro-2-methylphenol
GC-MS	EPA 8270C / 8270D	2,4-Dinitrophenol
GC-MS	EPA 8270C / 8270D	2,4-Dinitrotoluene
GC-MS	EPA 8270C / 8270D	2,6-Dinitrotoluene
GC-MS	EPA 8270C / 8270D	Di-n-octylphthalate
GC-MS	EPA 8270C / 8270D	Fluoranthene
GC-MS	EPA 8270C / 8270D	Fluorene
GC-MS	EPA 8270C / 8270D	Hexachlorobenzene
GC-MS	EPA 8270C / 8270D	Hexachlorobutadiene
GC-MS	EPA 8270C / 8270D	Hexachlorocyclopentadiene
GC-MS	EPA 8270C / 8270D	Hexachloroethane
GC-MS	EPA 8270C / 8270D	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 8270C / 8270D	Isophorone
GC-MS	EPA 8270C / 8270D	1-Methylnaphthalene
GC-MS	EPA 8270C / 8270D	2-Methylnaphthalene
GC-MS	EPA 8270C / 8270D	1-Methylphenanthrene
GC-MS	EPA 8270C / 8270D	2-Methylphenol

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	4-Methylphenol
GC-MS	EPA 8270C / 8270D	Naphthalene
GC-MS	EPA 8270C / 8270D	2-Nitroaniline
GC-MS	EPA 8270C / 8270D	3-Nitroaniline
GC-MS	EPA 8270C / 8270D	4-Nitroaniline
GC-MS	EPA 8270C / 8270D	Nitrobenzene
GC-MS	EPA 8270C / 8270D	2-Nitrophenol
GC-MS	EPA 8270C / 8270D	4-Nitrophenol
GC-MS	EPA 8270C / 8270D	n-Nitrosodimethylamine
GC-MS	EPA 8270C / 8270D	n-Nitroso-di-n-propylamine
GC-MS	EPA 8270C / 8270D	n-Nitrosodiphenylamine
GC-MS	EPA 8270C / 8270D	Pentachlorophenol
GC-MS	EPA 8270C / 8270D	Perylene
GC-MS	EPA 8270C / 8270D	Phenanthrene
GC-MS	EPA 8270C / 8270D	Phenol
GC-MS	EPA 8270C / 8270D	Pyrene
GC-MS	EPA 8270C / 8270D	Pyridine
GC-MS	EPA 8270C / 8270D	2,3,4,6-Tetrachlorophenol
GC-MS	EPA 8270C / 8270D	1,2,4-Trichlorobenzene
GC-MS	EPA 8270C / 8270D	2,3,4-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,3,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,4,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,4,6-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,3,5-Trimethylnaphthalene
GC-MS	EPA 8270C / 8270D	1,2,4,5-Tetrachlorobenzene
GC-MS	EPA 8270C / 8270D	1,3,5-Trinitrobenzene
GC-MS	EPA 8270C / 8270D	1,3-Dinitrobenzene
GC-MS	EPA 8270C / 8270D	1,4-Dioxane
GC-MS	EPA 8270C / 8270D	1,4-Naphthoquinone
GC-MS	EPA 8270C / 8270D	1-Chloronaphthalene
GC-MS	EPA 8270C / 8270D	1-Naphthylamine
GC-MS	EPA 8270C / 8270D	2,6-Dichlorophenol
GC-MS	EPA 8270C / 8270D	2-acetylaminofluorene
GC-MS	EPA 8270C / 8270D	2-Naphthylamine
GC-MS	EPA 8270C / 8270D	2-Picoline
GC-MS	EPA 8270C / 8270D	3,3-Dimethylbenzidine
GC-MS	EPA 8270C / 8270D	3,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D	3,5-Dimethylphenol
GC-MS	EPA 8270C / 8270D	3-Methylchlolanthrene
GC-MS	EPA 8270C / 8270D	4-Aminobiphenyl
GC-MS	EPA 8270C / 8270D	4-Nitroquinoline-N-oxide
GC-MS	EPA 8270C / 8270D	5-Nitro-o-toluidine
GC-MS	EPA 8270C / 8270D	7,12-Dimethylbenz(a)anthracene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	a,a-dimethylphenethylamine
GC-MS	EPA 8270C / 8270D	Acetophenone
GC-MS	EPA 8270C / 8270D	Aramite
GC-MS	EPA 8270C / 8270D	Atrazine
GC-MS	EPA 8270C / 8270D	Biphenyl
GC-MS	EPA 8270C / 8270D	Chlorobenzilate
GC-MS	EPA 8270C / 8270D	Diallate
GC-MS	EPA 8270C / 8270D	Dibenzo(a,j)acridine
GC-MS	EPA 8270C / 8270D	Dimethoate
GC-MS	EPA 8270C / 8270D	Dinoseb
GC-MS	EPA 8270C / 8270D	Diphenyl ether
GC-MS	EPA 8270C / 8270D	Disulfoton
GC-MS	EPA 8270C / 8270D	Ethyl methacrylate
GC-MS	EPA 8270C / 8270D	Ethyl methanesulfonate
GC-MS	EPA 8270C / 8270D	Ethyl parathion
GC-MS	EPA 8270C / 8270D	Famphur
GC-MS	EPA 8270C / 8270D	Hexachlorophene
GC-MS	EPA 8270C / 8270D	Hexachloropropene
GC-MS	EPA 8270C / 8270D	Isodrin
GC-MS	EPA 8270C / 8270D	Isosafrole
GC-MS	EPA 8270C / 8270D	kepone
GC-MS	EPA 8270C / 8270D	Methapyrilene
GC-MS	EPA 8270C / 8270D	Methyl methanesulfonate
GC-MS	EPA 8270C / 8270D	Methyl parathion
GC-MS	EPA 8270C / 8270D	N-nitrosodiethylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosodi-n-butylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosomethylethylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosomorpholine
GC-MS	EPA 8270C / 8270D	N-Nitrosopiperdine
GC-MS	EPA 8270C / 8270D	N-Nitrosopyrrolidine
GC-MS	EPA 8270C / 8270D	O,O,O-triethyl phosphorothi
GC-MS	EPA 8270C / 8270D	o-toluidine
GC-MS	EPA 8270C / 8270D	p-Dimethylaminoazobenze
GC-MS	EPA 8270C / 8270D	Pentachlorobenzene
GC-MS	EPA 8270C / 8270D	Pentachloroethane
GC-MS	EPA 8270C / 8270D	Pentachloronitrobenzene
GC-MS	EPA 8270C / 8270D	Phenacetin
GC-MS	EPA 8270C / 8270D	Phorate
GC-MS	EPA 8270C / 8270D	p-phenylenediamine
GC-MS	EPA 8270C / 8270D	Pronamide
GC-MS	EPA 8270C / 8270D	Safrole
GC-MS	EPA 8270C / 8270D	Sulfotepp
GC-MS	EPA 8270C / 8270D	Thionazin

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D SIM	Acenaphthene
GC-MS	EPA 8270C / 8270D SIM	Acenaphthylene
GC-MS	EPA 8270C / 8270D SIM	Anthracene
GC-MS	EPA 8270C / 8270D SIM	Azobenzene
GC-MS	EPA 8270C / 8270D SIM	Benzo(a)anthracene
GC-MS	EPA 8270C / 8270D SIM	benzo(a)pyrene
GC-MS	EPA 8270C / 8270D SIM	Benzo(b)fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Benzo(e)pyrene
GC-MS	EPA 8270C / 8270D SIM	Benzo(g,h,i)perylene
GC-MS	EPA 8270C / 8270D SIM	Benzo(k)fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Biphenyl
GC-MS	EPA 8270C / 8270D SIM	bis(2-chloroethyl)ether
GC-MS	EPA 8270C / 8270D SIM	bis(2-Ethylhexyl)phthalate
GC-MS	EPA 8270C / 8270D SIM	Carbazole
GC-MS	EPA 8270C / 8270D SIM	4-Chloro-3-methylphenol
GC-MS	EPA 8270C / 8270D SIM	2-Chlorophenol
GC-MS	EPA 8270C / 8270D SIM	Chrysene
GC-MS	EPA 8270C / 8270D SIM	Dibenzo(a,h)anthracene
GC-MS	EPA 8270C / 8270D SIM	2,4-Dichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,6-Dimethylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	2,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D SIM	Fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Fluorene
GC-MS	EPA 8270C / 8270D SIM	Hexachlorobenzene
GC-MS	EPA 8270C / 8270D SIM	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 8270C / 8270D SIM	1-Methylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	2-Methylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	1-Methylphenanthrene
GC-MS	EPA 8270C / 8270D SIM	Naphthalene
GC-MS	EPA 8270C / 8270D SIM	n-Nitrosodimethylamine
GC-MS	EPA 8270C / 8270D SIM	n-Nitroso-di-n-propylamine
GC-MS	EPA 8270C / 8270D SIM	Pentachlorophenol
GC-MS	EPA 8270C / 8270D SIM	Perylene
GC-MS	EPA 8270C / 8270D SIM	Phenanthrene
GC-MS	EPA 8270C / 8270D SIM	Phenol
GC-MS	EPA 8270C / 8270D SIM	Pyrene
GC-MS	EPA 8270C / 8270D SIM	2,4,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,4,6-Trichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,3,5-Trimethylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	1,4-Dioxane
GC-MS	EPA 8270C / 8270D SIM	Butylbenzylphthalate
GC-MS	EPA 8270C / 8270D SIM	Diethylphthalate
GC-MS	EPA 8270C / 8270D SIM	Dimethylphthalate

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D SIM	Di-n-butylphthalate
GC-MS	EPA 8270C / 8270D SIM	Di-n-octylphthalate
HPLC	EPA 8310	Acenaphthene
HPLC	EPA 8310	Acenaphthylene
HPLC	EPA 8310	Anthracene
HPLC	EPA 8310	Benzo(a)anthracene
HPLC	EPA 8310	Benzo(a)pyrene
HPLC	EPA 8310	Benzo(b)fluoranthene
HPLC	EPA 8310	Benzo(g,h,i)perylene
HPLC	EPA 8310	Benzo(k)fluoranthene
HPLC	EPA 8310	Chrysene
HPLC	EPA 8310	Dibenzo(a,h)anthracene
HPLC	EPA 8310	Fluoranthene
HPLC	EPA 8310	Fluorene
HPLC	EPA 8310	Indeno(1,2,3-cd)pyrene
HPLC	EPA 8310	1-Methylnaphthalene
HPLC	EPA 8310	2-Methylnaphthalene
HPLC	EPA 8310	Naphthalene
HPLC	EPA 8310	Phenanthrene
HPLC	EPA 8310	Pyrene
HPLC	EPA 8330A / 8330 B	HMX
HPLC	EPA 8330A / 8330 B	RDX
HPLC	EPA 8330A / 8330 B	1,3,5-TNB
HPLC	EPA 8330A / 8330 B	1,3-DNB
HPLC	EPA 8330A / 8330 B	Tetryl
HPLC	EPA 8330A / 8330 B	Nitrobenzene
HPLC	EPA 8330A / 8330 B	2,4,6-TNT
HPLC	EPA 8330A / 8330 B	4-AM-2,6-DNT
HPLC	EPA 8330A / 8330 B	2-AM-4,6-DNT
HPLC	EPA 8330A / 8330 B	2,6-DNT
HPLC	EPA 8330A / 8330 B	2,4-DNT
HPLC	EPA 8330A / 8330 B	2-Nitrotoluene
HPLC	EPA 8330A / 8330 B	4-Nitrotoluene
HPLC	EPA 8330A / 8330 B	3-Nitrotoluene
HPLC	EPA 8330A	3,5-Dinitroaniline
HPLC	EPA 8330A	2,4-Diamino-6-nitrotoluene
HPLC	EPA 8330A	2,6-Diamino-4-nitrotoluene
HPLC	EPA 8330A	Picric Acid
HPLC	EPA 8332	Nitroglycerine
HPLC	EPA 8332	PETN
Spectrometric	EPA 9014	Cyanide
Electrode	EPA 9040 B / 9040C	pH
IC	EPA 9056 / 9056A	Bromate

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
IC	EPA 9056 / 9056A	Bromide
IC	EPA 9056 / 9056A	Chloride
IC	EPA 9056 / 9056A	Fluoride
IC	EPA 9056 / 9056A	Nitrate
IC	EPA 9056 / 9056A	Nitrite
IC	EPA 9056 / 9056A	Phosphate
IC	EPA 9056 / 9056A	Sulfate
IC	EPA 9056 / 9056A	Chlorate
Combustion-IR	EPA 9060A	TOC
Spectrometric	EPA 9065	Phenols
Gravimetric	EPA 9070A	Oil & Grease
Gravimetric	EPA 9071B	Oil & Grease
GC	RSK175	Methane
GC	RSK175	Acetylene
GC	RSK175	Ethylene
GC	RSK175	Ethane
GC	RSK175	Propane
GC	RSK175	Carbon dioxide
Spectrometric	SM 4500-NH3C	Ammonia
Spectrometric	SM 4500-NH3F	Ammonia
Spectrometric	SM 4500-NOrgC	TKN
Spectrometric	SM 4500-PE	Phosphorus
Turbidimetric	SM 2130B	Turbidity
Titrimetric	SM 2310B	Acidity
Titrimetric	SM 2320B	Alkalinity
Titrimetric	SM 2340C	Hardness
Platinum Electrode	SM 2510B	Specific Conductance
Gravimetric	SM 2540C	TDS
Gravimetric	SM 2540D	TSS
Gravimetric	SM 2540B	Total Residue
Spectrometric	SM 3500-FeB	Ferrous iron
IC	SM 4110B	Bromate
IC	SM 4110B	Bromide
IC	SM 4110B	Chloride
IC	SM 4110B	Fluoride
IC	SM 4110B	Nitrate
IC	SM 4110B	Nitrite
IC	SM 4110B	Phosphate
IC	SM 4110B	Sulfate
IC	SM 4110B	Chlorate
Titrimetric	SM 4500-Cl-B	Chloride
Titrimetric	SM 4500-Cl B	Total Residual Chlorine
Spectrometric	SM 4500CNE	Cyanide

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
Electrode	SM 4500-FC	Fluoride
Electrode	SM 4500 HB	pH
Spectrometric	SM4500-NO2B	Nitrite-N
Spectrometric	SM4500-NO3E	Nitrate-N
Spectrometric	SM4500PE	Ortho-phosphate
Spectrometric	SM4500-PE(PB5)	Phosphorus
Spectrometric	SM4500-S2D	Sulfide
Titrimetric	SM4500-S2F	Sulfide
Spectrometric	SM4500-SiO2C	Silica
Electrode	SM5210B	BOD
Spectrometric	SM 5220D	COD
Combustion-IR	SM 5310B	TOC
Spectrometric	SM5540C	Surfactants (MBAS)
Distillation	EPA 9010C	Cyanide
MicroDistillation	QuickChem 10-204-00-1-X	Cyanide
ICP/ICP-MS	SM2340B	Hardness
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Purge & Trap	EPA 5030B / 5030C	Volatiles Prep
Acid Digestion	EPA 3005A / EPA 3010A / EPA 200.8 / EPA 200.7	Metals Prep
Continuous Liquid-Liquid	EPA 3520C	Organic Extraction
Waste Dilution	EPA 3580A	Organic Extraction
TCLP	EPA 1311	Leaching
SPLP	EPA 1312	Leaching

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
Platinum Electrode	EPA 120.1	Specific Conductance
Electrode	EPA 150.1	pH
Gravimetric	EPA 160.1	TDS
Gravimetric	EPA 160.2	TSS
Gravimetric	EPA 160.3	Total Residue
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Chromium

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium
ICP-MS	EPA 200.8	Zinc
IC	EPA 218.6	Hexavalent Chromium
Cold Vapor	EPA 245.1	Mercury
IC	EPA 300.0	Bromate
IC	EPA 300.0	Bromide
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Phosphate
IC	EPA 300.0	Sulfate
IC	EPA 300.0	Chlorate
IC	EPA 300M	Acetate
IC	EPA 300M	Butyrate
IC	EPA 300M	Lactate
IC	EPA 300M	Propionate
IC	EPA 300M	Pyruvate
IC	EPA 314.0	Perchlorate
Spectrometric	EPA 335.2	Cyanide
Spectrometric	EPA 350.2	Ammonia
Spectrometric	EPA 351.3	TKN
Spectrometric	EPA 352.1	Nitrate-N

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
Spectrometric	EPA 353.3	Nitrate-N
Spectrometric	EPA 354.1	Nitrite-N
Spectrometric	EPA 365.2	Ortho-phosphate
Spectrometric	EPA 365.2	Phosphorus
Spectrometric	EPA 370.1	Silica
Titrimetric	EPA 376.1	Sulfide
Spectrometric	EPA 410.4	COD
Combustion-IR	EPA 415.1	TOC
Spectrometric	EPA 420.1	Phenols
GC	EPA 504.1	DBCP
GC	EPA 504.1	EDB
GC-MS	EPA 524.2	Acetone
GC-MS	EPA 524.2	Benzene
GC-MS	EPA 524.2	Bromobenzene
GC-MS	EPA 524.2	Bromochloromethane
GC-MS	EPA 524.2	Bromodichloromethane
GC-MS	EPA 524.2	Bromoform
GC-MS	EPA 524.2	Bromomethane
GC-MS	EPA 524.2	tert-Butyl alcohol
GC-MS	EPA 524.2	2-Butanone (MEK)
GC-MS	EPA 524.2	n-Butylbenzene
GC-MS	EPA 524.2	sec-Butylbenzene
GC-MS	EPA 524.2	tert-Butylbenzene
GC-MS	EPA 524.2	Carbon disulfide
GC-MS	EPA 524.2	Carbon tetrachloride
GC-MS	EPA 524.2	Chlorobenzene
GC-MS	EPA 524.2	Chloroethane
GC-MS	EPA 524.2	Chloroform
GC-MS	EPA 524.2	Chloromethane
GC-MS	EPA 524.2	2-Chlorotoluene
GC-MS	EPA 524.2	4-Chlorotoluene
GC-MS	EPA 524.2	Dibromochloromethane
GC-MS	EPA 524.2	1,2-Dibromo-3-chloropropane
GC-MS	EPA 524.2	1,2-Dibromoethane
GC-MS	EPA 524.2	Dibromomethane
GC-MS	EPA 524.2	1,1-Dichloroethane
GC-MS	EPA 524.2	1,2-Dichloroethane
GC-MS	EPA 524.2	1,2-Dichlorobenzene
GC-MS	EPA 524.2	1,3-Dichlorobenzene
GC-MS	EPA 524.2	1,4-Dichlorobenzene
GC-MS	EPA 524.2	Dichlorodifluoromethane

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 524.2	1,1-Dichloroethene
GC-MS	EPA 524.2	cis-1,2-Dichloroethene
GC-MS	EPA 524.2	trans-1,2-Dichloroethene
GC-MS	EPA 524.2	1,1-Dichloropropene
GC-MS	EPA 524.2	1,2-Dichloropropane
GC-MS	EPA 524.2	1,3-Dichloropropane
GC-MS	EPA 524.2	2,2-Dichloropropane
GC-MS	EPA 524.2	cis-1,3-Dichloropropene
GC-MS	EPA 524.2	trans-1,3-Dichloropropene
GC-MS	EPA 524.2	tert-Butyl ethyl ether (ETBE)
GC-MS	EPA 524.2	Ethylbenzene
GC-MS	EPA 524.2	2-Hexanone (MBK)
GC-MS	EPA 524.2	Hexachlorobutadiene
GC-MS	EPA 524.2	Isopropyl ether (DIPE)
GC-MS	EPA 524.2	Isopropylbenzene
GC-MS	EPA 524.2	p-Isopropyltoluene
GC-MS	EPA 524.2	Methylene Chloride
GC-MS	EPA 524.2	4-Methyl-2-pentanone (MIBK)
GC-MS	EPA 524.2	tert-Butyl methyl ether
GC-MS	EPA 524.2	Naphthalene
GC-MS	EPA 524.2	n-Propylbenzene
GC-MS	EPA 524.2	Styrene
GC-MS	EPA 524.2	tert-Amyl methyl ether (TAME)
GC-MS	EPA 524.2	1,1,1,2-Tetrachloroethane
GC-MS	EPA 524.2	1,1,2,2-Tetrachloroethane
GC-MS	EPA 524.2	Tetrachloroethene
GC-MS	EPA 524.2	Toluene
GC-MS	EPA 524.2	1,1,1-Trichloroethane
GC-MS	EPA 524.2	1,1,2-Trichloroethane
GC-MS	EPA 524.2	1,2,3-Trichlorobenzene
GC-MS	EPA 524.2	1,2,4-Trichlorobenzene
GC-MS	EPA 524.2	Trichloroethene
GC-MS	EPA 524.2	Trichlorofluoromethane
GC-MS	EPA 524.2	1,2,3-Trichloropropane
GC-MS	EPA 524.2	1,1,2-Trichloro-1,2,2-trifluoroethane
GC-MS	EPA 524.2	1,2,4-Trimethylbenzene
GC-MS	EPA 524.2	1,3,5-Trimethylbenzene
GC-MS	EPA 524.2	Vinyl Chloride
GC-MS	EPA 524.2	m-Xylene & p-xylene
GC-MS	EPA 524.2	o-Xylene
Titrimetric	SM 2320B	Alkalinity

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC-MS	EPA 6850	Perchlorate
ICP/ICP-MS by Calculation	SM 2340B	Hardness
Titrimetric	SM 2340C	Hardness
Platinum Electrode	SM 2510B	Specific Conductance
Gravimetric	SM 2540B	Total Residue
Gravimetric	SM 2540C	TDS
Gravimetric	SM 2540D	TSS
Spectrometric	SM 3500- FeB	Ferrous Iron
Spectrometric	SM 4500-CNE	Cyanide
Electrode	SM 4500 HB	pH
Spectrometric	SM 4500-NH3C	Ammonia
Spectrometric	SM 4500-NH3F	Ammonia
Spectrometric	SM 4500-NO2B	Nitrite-N
Spectrometric	SM 4500-NO3E	Nitrate-N
Spectrometric	SM 4500-NOrgC	TKN
Spectrometric	SM 4500-PE	Ortho-phosphate
Spectrometric	SM 4500-PE(PB5)	Phosphorus
Titrimetric	SM 4500-S2F	Sulfide
Spectrometric	SM 4500-SiO2C	Silica
Spectrometric	SM 5220D	COD
Combustion-IR	SM 5310B	TOC
Spectrometric	SM 5540C	Surfactants
MicroDistillation	QuickChem 10-204-00-1-X	Cyanide

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	AK101	GRO
GC	AK102	DRO
GC	AK103	RRO
GC	AZ8015	DRO (C10-C22)
GC	AZ8015	ORO (C22-C32)
GC	RSK175	Methane
GC	RSK175	Acetylene
GC	RSK175	Ethylene
GC	RSK175	Ethane
GC	RSK175	Propane
GC	RSK175	Carbon dioxide
Spectrometric	SM4500-NH3C	Ammonia
Spectrometric	SM4500-NH3F	Ammonia
Spectrometric	SM4500-NOrgC	TKN

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
Spectrometric	SM4500-PE(PB5)	Phosphorus
Titrimetric	Walkley Black	TOC
Electrode	EPA 9045C / 9045D	pH
Spectrometric	EPA 9065	Phenols
Penskey-Martens	EPA 1010/ 1010A	Ignitability
ICP	EPA 6010B / 6010C	Aluminum
ICP	EPA 6010B / 6010C	Antimony
ICP	EPA 6010B / 6010C	Arsenic
ICP	EPA 6010B / 6010C	Barium
ICP	EPA 6010B / 6010C	Beryllium
ICP	EPA 6010B / 6010C	Boron
ICP	EPA 6010B / 6010C	Cadmium
ICP	EPA 6010B / 6010C	Calcium
ICP	EPA 6010B / 6010C	Chromium
ICP	EPA 6010B / 6010C	Cobalt
ICP	EPA 6010B / 6010C	Copper
ICP	EPA 6010B / 6010C	Iron
ICP	EPA 6010B / 6010C	Lead
ICP	EPA 6010B / 6010C	Lithium
ICP	EPA 6010B / 6010C	Magnesium
ICP	EPA 6010B / 6010C	Manganese
ICP	EPA 6010B / 6010C	Molybdenum
ICP	EPA 6010B / 6010C	Nickel
ICP	EPA 6010B / 6010C	Potassium
ICP	EPA 6010B / 6010C	Selenium
ICP	EPA 6010B / 6010C	Silver
ICP	EPA 6010B / 6010C	Sodium
ICP	EPA 6010B / 6010C	Strontium
ICP	EPA 6010B / 6010C	Thallium
ICP	EPA 6010B / 6010C	Tin
ICP	EPA 6010B / 6010C	Titanium
ICP	EPA 6010B / 6010C	Vanadium
ICP	EPA 6010B / 6010C	Zinc
IPC-MS	EPA 6020A	Aluminum
IPC-MS	EPA 6020A	Antimony
IPC-MS	EPA 6020A	Arsenic
IPC-MS	EPA 6020A	Barium
IPC-MS	EPA 6020A	Beryllium
IPC-MS	EPA 6020A	Boron
IPC-MS	EPA 6020A	Cadmium
IPC-MS	EPA 6020A	Calcium
IPC-MS	EPA 6020A	Chromium
IPC-MS	EPA 6020A	Cobalt

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
IPC-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Lithium
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Nickel
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Zinc
HPLC-MS	EPA 6850	Perchlorate
Spectrometric	EPA 7196A	Hex. Chromium
IC	EPA 7199	Hex. Chromium
Cold-Vapor	EPA 7471A / 7471B	Mercury
GC	EPA 8011	DBCP
GC	EPA 8011	EDB
GC	EPA 8015B / 8015C / 8015D	Gasoline
GC	EPA 8015B / 8015C / 8015D	Diesel
GC	EPA 8015B / 8015C / 8015D	Motor Oil
GC	EPA 8015B / 8015C / 8015D	JP5
GC	EPA 8015B / 8015C	Ethanol
GC	EPA 8015B / 8015C	Isopropanol
GC	EPA 8015B / 8015C	Diethylene Glycol
GC	EPA 8015B / 8015C	Ethylene Glycol
GC	EPA 8015B / 8015C	Triethylene Glycol
GC	EPA 8015B / 8015C / 8015D	JP4
GC	EPA 8015B / 8015C	Methanol
GC	EPA 8015B / 8015C	Propylene Glycol
GC	EPA 8081A / 8081B	Aldrin
GC	EPA 8081A / 8081B	alpha-BHC
GC	EPA 8081A / 8081B	beta-BHC
GC	EPA 8081A / 8081B	delta-BHC
GC	EPA 8081A / 8081B	gamma-BHC (Lindane)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8081A / 8081B	DDD (4,4)
GC	EPA 8081A / 8081B	DDE (4,4)
GC	EPA 8081A / 8081B	DDT (4,4)
GC	EPA 8081A / 8081B	Dieldrin
GC	EPA 8081A / 8081B	Endosulfan I
GC	EPA 8081A / 8081B	Endosulfan II
GC	EPA 8081A / 8081B	Endosulfan sulfate
GC	EPA 8081A / 8081B	Endrin
GC	EPA 8081A / 8081B	Endrin Aldehyde
GC	EPA 8081A / 8081B	Heptachlor
GC	EPA 8081A / 8081B	Heptachlor epoxide
GC	EPA 8081A / 8081B	Methoxychlor
GC	EPA 8081A / 8081B	alpha-Chlordane
GC	EPA 8081A / 8081B	gamma-Chlordane
GC	EPA 8081A / 8081B	Endrin Ketone
GC	EPA 8081A / 8081B	Toxaphene
GC	EPA 8081A / 8081B	Technical Chlordane
GC	EPA 8081A / 8081B	cis-Nonachlor
GC	EPA 8081A / 8081B	DDD (2,4)
GC	EPA 8081A / 8081B	DDE (2,4)
GC	EPA 8081A / 8081B	DDT (2,4)
GC	EPA 8081A / 8081B	Mirex
GC	EPA 8081A / 8081B	Oxychlordane
GC	EPA 8081A / 8081B	trans-Nonachlor
GC	EPA 8082 / 8082A	PCB1016
GC	EPA 8082 / 8082A	PCB1221
GC	EPA 8082 / 8082A	PCB1232
GC	EPA 8082 / 8082A	PCB1242
GC	EPA 8082 / 8082A	PCB1248
GC	EPA 8082 / 8082A	PCB1254
GC	EPA 8082 / 8082A	PCB1260
GC	EPA 8082 / 8082A	PCB1262
GC	EPA 8082 / 8082A	PCB1268
GC	EPA 8082 / 8082A	PCB 8
GC	EPA 8082 / 8082A	PCB 18
GC	EPA 8082 / 8082A	PCB 28
GC	EPA 8082 / 8082A	PCB 44
GC	EPA 8082 / 8082A	PCB 52
GC	EPA 8082 / 8082A	PCB 66
GC	EPA 8082 / 8082A	PCB 77
GC	EPA 8082 / 8082A	PCB 81
GC	EPA 8082 / 8082A	PCB 101
GC	EPA 8082 / 8082A	PCB 105

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8082 / 8082A	PCB 110
GC	EPA 8082 / 8082A	PCB 114
GC	EPA 8082 / 8082A	PCB 118
GC	EPA 8082 / 8082A	PCB 123
GC	EPA 8082 / 8082A	PCB 126
GC	EPA 8082 / 8082A	PCB 128
GC	EPA 8082 / 8082A	PCB 138
GC	EPA 8082 / 8082A	PCB 153
GC	EPA 8082 / 8082A	PCB 156
GC	EPA 8082 / 8082A	PCB 157
GC	EPA 8082 / 8082A	PCB 167
GC	EPA 8082 / 8082A	PCB 169
GC	EPA 8082 / 8082A	PCB 170
GC	EPA 8082 / 8082A	PCB 180
GC	EPA 8082 / 8082A	PCB 187
GC	EPA 8082 / 8082A	PCB 189
GC	EPA 8082 / 8082A	PCB 195
GC	EPA 8082 / 8082A	PCB 206
GC	EPA 8082 / 8082A	PCB 209
GC	EPA 8141A / 8141B	Azinphos-methyl
GC	EPA 8141A / 8141B	Bolstar
GC	EPA 8141A / 8141B	Chlorpyrifos
GC	EPA 8141A / 8141B	Coumaphos
GC	EPA 8141A / 8141B	Demeton
GC	EPA 8141A / 8141B	Diazinon
GC	EPA 8141A / 8141B	Dichlorvos
GC	EPA 8141A / 8141B	Disulfoton
GC	EPA 8141A / 8141B	Ethoprop
GC	EPA 8141A / 8141B	Fensulfothion
GC	EPA 8141A / 8141B	Fenthion
GC	EPA 8141A / 8141B	Merphos
GC	EPA 8141A / 8141B	Mevinphos
GC	EPA 8141A / 8141B	Naled
GC	EPA 8141A / 8141B	Methyl Parathion
GC	EPA 8141A / 8141B	Phorate
GC	EPA 8141A / 8141B	Ronnel
GC	EPA 8141A / 8141B	Stirophos
GC	EPA 8141A / 8141B	Tokuthion
GC	EPA 8141A / 8141B	Trichloronate
GC	EPA 8141A / 8141B	Dimethoate
GC	EPA 8141A / 8141B	EPN
GC	EPA 8141A / 8141B	Famphur
GC	EPA 8141A / 8141B	Malathion

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC	EPA 8141A / 8141B	Ethyl Parathion
GC	EPA 8141A / 8141B	O,O,O-Triethylphosphorothioate
GC	EPA 8141A / 8141B	Sulfotepp
GC	EPA 8141A / 8141B	Thionazin
GC	EPA 8141A / 8141B	Tributyl Phosphate
GC-MS	EPA 8260B / 8260C	Acetone
GC-MS	EPA 8260B / 8260C	Acrolein
GC-MS	EPA 8260B / 8260C	Acrylonitrile
GC-MS	EPA 8260B / 8260C	Benzene
GC-MS	EPA 8260B / 8260C	Bromobenzene
GC-MS	EPA 8260B / 8260C	Bromochloromethane
GC-MS	EPA 8260B / 8260C	Bromodichloromethane
GC-MS	EPA 8260B / 8260C	Bromoform
GC-MS	EPA 8260B / 8260C	Bromomethane
GC-MS	EPA 8260B / 8260C	tert-Butyl alcohol
GC-MS	EPA 8260B / 8260C	2-Butanone (MEK)
GC-MS	EPA 8260B / 8260C	n-Butylbenzene
GC-MS	EPA 8260B / 8260C	sec-Butylbenzene
GC-MS	EPA 8260B / 8260C	tert-Butylbenzene
GC-MS	EPA 8260B / 8260C	Carbon disulfide
GC-MS	EPA 8260B / 8260C	Carbon tetrachloride
GC-MS	EPA 8260B / 8260C	Chlorobenzene
GC-MS	EPA 8260B / 8260C	2-Chloroethyl vinyl ether
GC-MS	EPA 8260B / 8260C	Chloroethane
GC-MS	EPA 8260B / 8260C	Chloroform
GC-MS	EPA 8260B / 8260C	1-Chlorohexane
GC-MS	EPA 8260B / 8260C	Chloromethane
GC-MS	EPA 8260B / 8260C	2-Chlorotoluene
GC-MS	EPA 8260B / 8260C	4-Chlorotoluene
GC-MS	EPA 8260B / 8260C	Isopropyl ether (DIPE)
GC-MS	EPA 8260B / 8260C	Dibromochloromethane
GC-MS	EPA 8260B / 8260C	1,2-Dibromo-3-chloropropane
GC-MS	EPA 8260B / 8260C	1,2-Dibromoethane
GC-MS	EPA 8260B / 8260C	Dibromomethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloroethane
GC-MS	EPA 8260B / 8260C	1,2-Dichloroethane
GC-MS	EPA 8260B / 8260C	1,2-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	1,3-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	trans-1,4-Dichloro-2-Butene
GC-MS	EPA 8260B / 8260C	1,4-Dichlorobenzene
GC-MS	EPA 8260B / 8260C	Dichlorodifluoromethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloroethene
GC-MS	EPA 8260B / 8260C	cis-1,2-Dichloroethene

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8260B / 8260C	trans-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C	Dichlorofluoromethane
GC-MS	EPA 8260B / 8260C	1,1-Dichloropropene
GC-MS	EPA 8260B / 8260C	1,2-Dichloropropane
GC-MS	EPA 8260B / 8260C	1,3-Dichloropropane
GC-MS	EPA 8260B / 8260C	2,2-Dichloropropane
GC-MS	EPA 8260B / 8260C	cis-1,3-Dichloropropene
GC-MS	EPA 8260B / 8260C	trans-1,3-Dichloropropene
GC-MS	EPA 8260B / 8260C	tert-Butyl ethyl ether (ETBE)
GC-MS	EPA 8260B / 8260C	Ethyl Methacrylate
GC-MS	EPA 8260B / 8260C	Ethylbenzene
GC-MS	EPA 8260B / 8260C	2-Hexanone (MBK)
GC-MS	EPA 8260B / 8260C	Hexachlorobutadiene
GC-MS	EPA 8260B / 8260C	Iodomethane
GC-MS	EPA 8260B / 8260C	Isopropylbenzene
GC-MS	EPA 8260B / 8260C	p-Isopropyltoluene
GC-MS	EPA 8260B / 8260C	Methylene Chloride
GC-MS	EPA 8260B / 8260C	4-Methyl-2-pentanone (MIBK)
GC-MS	EPA 8260B / 8260C	tert-Butyl methyl ether
GC-MS	EPA 8260B / 8260C	Naphthalene
GC-MS	EPA 8260B / 8260C	n-Propylbenzene
GC-MS	EPA 8260B / 8260C	Styrene
GC-MS	EPA 8260B / 8260C	tert-Amyl methyl ether (TAME)
GC-MS	EPA 8260B / 8260C	1,1,1,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C	1,1,2,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C	Tetrachloroethene
GC-MS	EPA 8260B / 8260C	Toluene
GC-MS	EPA 8260B / 8260C	1,1,1-Trichloroethane
GC-MS	EPA 8260B / 8260C	1,1,2-Trichloroethane
GC-MS	EPA 8260B / 8260C	1,2,3-Trichlorobenzene
GC-MS	EPA 8260B / 8260C	1,2,4-Trichlorobenzene
GC-MS	EPA 8260B / 8260C	Trichloroethene
GC-MS	EPA 8260B / 8260C	Trichlorofluoromethane
GC-MS	EPA 8260B / 8260C	1,2,3-Trichloropropane
GC-MS	EPA 8260B / 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC-MS	EPA 8260B / 8260C	1,2,4-Trimethylbenzene
GC-MS	EPA 8260B / 8260C	1,3,5-Trimethylbenzene
GC-MS	EPA 8260B / 8260C	Vinyl Acetate
GC-MS	EPA 8260B / 8260C	Vinyl Chloride
GC-MS	EPA 8260B / 8260C	m-Xylene & p-xylene
GC-MS	EPA 8260B / 8260C	o-Xylene
GC-MS	EPA 8260B / 8260C	2-Butanol
GC-MS	EPA 8260B / 8260C	Cyclohexane

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8260B / 8260C	1,4-Dioxane
GC-MS	EPA 8260B / 8260C	2-Chloro-1,1,1-trifluoroethane
GC-MS	EPA 8260B / 8260C	Chlorotrifluoroethylene
GC-MS	EPA 8260B / 8260C	cis-1,4-Dichloro-2-butene
GC-MS	EPA 8260B / 8260C	Ethanol
GC-MS	EPA 8260B / 8260C	Ethyl Methacrylate
GC-MS	EPA 8260B / 8260C	Isobutyl Alcohol
GC-MS	EPA 8260B / 8260C	Methacrylonitrile
GC-MS	EPA 8260B / 8260C	Methyl Methacrylate
GC-MS	EPA 8260B / 8260C	Pentachloroethane
GC-MS	EPA 8260B / 8260C	Propionitrile
GC-MS	EPA 8260B / 8260C	Sec-Propyl alcohol
GC-MS	EPA 8260B / 8260C	Tetrahydrofuran
GC-MS	EPA 8260B / 8260C	trans-1,4-Dichloro-2-butene
GC-MS	EPA 8260B / 8260C SIM	Benzene
GC-MS	EPA 8260B / 8260C SIM	Carbon tetrachloride
GC-MS	EPA 8260B / 8260C SIM	Chloroform
GC-MS	EPA 8260B / 8260C SIM	Chloromethane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dibromo-3-chloropropane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dibromoethane
GC-MS	EPA 8260B / 8260C SIM	1,2-Dichloroethane
GC-MS	EPA 8260B / 8260C SIM	1,1-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	cis-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	trans-1,2-Dichloroethene
GC-MS	EPA 8260B / 8260C SIM	1,1,2,2-Tetrachloroethane
GC-MS	EPA 8260B / 8260C SIM	Tetrachloroethene
GC-MS	EPA 8260B / 8260C SIM	1,1,1-Trichloroethane
GC-MS	EPA 8260B / 8260C SIM	1,1,2-Trichloroethane
GC-MS	EPA 8260B / 8260C SIM	Trichloroethene
GC-MS	EPA 8260B / 8260C SIM	1,2,3-Trichloropropane
GC-MS	EPA 8260B / 8260C SIM	Vinyl Chloride
GC-MS	EPA 8260B / 8260C SIM	1,4-Dioxane
GC-MS	EPA 8270C / 8270D	Acenaphthene
GC-MS	EPA 8270C / 8270D	Acenaphthylene
GC-MS	EPA 8270C / 8270D	Aniline
GC-MS	EPA 8270C / 8270D	Anthracene
GC-MS	EPA 8270C / 8270D	Azobenzene
GC-MS	EPA 8270C / 8270D	Benzidine
GC-MS	EPA 8270C / 8270D	Benzo(a)anthracene
GC-MS	EPA 8270C / 8270D	benzo(a)pyrene
GC-MS	EPA 8270C / 8270D	Benzo(b)fluoranthene
GC-MS	EPA 8270C / 8270D	Benzo(e)pyrene
GC-MS	EPA 8270C / 8270D	Benzo(g,h,i)perylene

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	Benzo(k)fluoranthene
GC-MS	EPA 8270C / 8270D	Benzoic Acid
GC-MS	EPA 8270C / 8270D	Benzyl Alcohol
GC-MS	EPA 8270C / 8270D	Biphenyl
GC-MS	EPA 8270C / 8270D	bis(2-chloroethoxy)methane
GC-MS	EPA 8270C / 8270D	bis(2-chloroethyl)ether
GC-MS	EPA 8270C / 8270D	bis(2-chloroisopropyl)ether
GC-MS	EPA 8270C / 8270D	bis(2-Ethylhexyl)adipate
GC-MS	EPA 8270C / 8270D	bis(2-Ethylhexyl)phthalate
GC-MS	EPA 8270C / 8270D	4-Bromophenyl-phenylether
GC-MS	EPA 8270C / 8270D	Butylbenzylphthalate
GC-MS	EPA 8270C / 8270D	Carbazole
GC-MS	EPA 8270C / 8270D	4-Chloro-3-methylphenol
GC-MS	EPA 8270C / 8270D	4-Chloroaniline
GC-MS	EPA 8270C / 8270D	2-Chloronaphthalene
GC-MS	EPA 8270C / 8270D	2-Chlorophenol
GC-MS	EPA 8270C / 8270D	4-Chlorophenyl-phenylether
GC-MS	EPA 8270C / 8270D	Chrysene
GC-MS	EPA 8270C / 8270D	Dibenzo(a,h)anthracene
GC-MS	EPA 8270C / 8270D	Dibenzofuran
GC-MS	EPA 8270C / 8270D	1,2-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	1,3-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	1,4-Dichlorobenzene
GC-MS	EPA 8270C / 8270D	3,3'-Dichlorobenzidine
GC-MS	EPA 8270C / 8270D	2,4-Dichlorophenol
GC-MS	EPA 8270C / 8270D	Diethylphthalate
GC-MS	EPA 8270C / 8270D	2,6-Dimethylnaphthalene
GC-MS	EPA 8270C / 8270D	2,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D	Dimethylphthalate
GC-MS	EPA 8270C / 8270D	Di-n-butylphthalate
GC-MS	EPA 8270C / 8270D	4,6-Dinitro-2-methylphenol
GC-MS	EPA 8270C / 8270D	2,4-Dinitrophenol
GC-MS	EPA 8270C / 8270D	2,4-Dinitrotoluene
GC-MS	EPA 8270C / 8270D	2,6-Dinitrotoluene
GC-MS	EPA 8270C / 8270D	Di-n-octylphthalate
GC-MS	EPA 8270C / 8270D	Fluoranthene
GC-MS	EPA 8270C / 8270D	Fluorene
GC-MS	EPA 8270C / 8270D	Hexachlorobenzene
GC-MS	EPA 8270C / 8270D	Hexachlorobutadiene
GC-MS	EPA 8270C / 8270D	Hexachlorocyclopentadiene
GC-MS	EPA 8270C / 8270D	Hexachloroethane
GC-MS	EPA 8270C / 8270D	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 8270C / 8270D	Isophorone

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	1-Methylnaphthalene
GC-MS	EPA 8270C / 8270D	2-Methylnaphthalene
GC-MS	EPA 8270C / 8270D	1-Methylphenanthrene
GC-MS	EPA 8270C / 8270D	2-Methylphenol
GC-MS	EPA 8270C / 8270D	4-Methylphenol
GC-MS	EPA 8270C / 8270D	Naphthalene
GC-MS	EPA 8270C / 8270D	2-Nitroaniline
GC-MS	EPA 8270C / 8270D	3-Nitroaniline
GC-MS	EPA 8270C / 8270D	4-Nitroaniline
GC-MS	EPA 8270C / 8270D	Nitrobenzene
GC-MS	EPA 8270C / 8270D	2-Nitrophenol
GC-MS	EPA 8270C / 8270D	4-Nitrophenol
GC-MS	EPA 8270C / 8270D	n-Nitrosodimethylamine
GC-MS	EPA 8270C / 8270D	n-Nitroso-di-n-propylamine
GC-MS	EPA 8270C / 8270D	n-Nitrosodiphenylamine
GC-MS	EPA 8270C / 8270D	Pentachlorophenol
GC-MS	EPA 8270C / 8270D	Perylene
GC-MS	EPA 8270C / 8270D	Phenanthrene
GC-MS	EPA 8270C / 8270D	Phenol
GC-MS	EPA 8270C / 8270D	Pyrene
GC-MS	EPA 8270C / 8270D	Pyridine
GC-MS	EPA 8270C / 8270D	2,3,4,6-Tetrachlorophenol
GC-MS	EPA 8270C / 8270D	1,2,4-Trichlorobenzene
GC-MS	EPA 8270C / 8270D	2,3,4-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,3,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,4,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,4,6-Trichlorophenol
GC-MS	EPA 8270C / 8270D	2,3,5-Trimethylnaphthalene
GC-MS	EPA 8270C / 8270D	1,2,4,5-Tetrachlorobenzene
GC-MS	EPA 8270C / 8270D	1,3,5-Trinitrobenzene
GC-MS	EPA 8270C / 8270D	1,3-Dinitrobenzene
GC-MS	EPA 8270C / 8270D	1,4-Dioxane
GC-MS	EPA 8270C / 8270D	1,4-Naphthoquinone
GC-MS	EPA 8270C / 8270D	1-Chloronaphthalene
GC-MS	EPA 8270C / 8270D	1-Naphthylamine
GC-MS	EPA 8270C / 8270D	2,6-Dichlorophenol
GC-MS	EPA 8270C / 8270D	2-acetylaminofluorene
GC-MS	EPA 8270C / 8270D	2-Naphthylamine
GC-MS	EPA 8270C / 8270D	2-Picoline
GC-MS	EPA 8270C / 8270D	3,3-Dimethylbenzidine
GC-MS	EPA 8270C / 8270D	3,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D	3,5-Dimethylphenol
GC-MS	EPA 8270C / 8270D	3-Methylcholanthrene

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	4-Aminobiphenyl
GC-MS	EPA 8270C / 8270D	4-Nitroquinoline-N-oxide
GC-MS	EPA 8270C / 8270D	5-Nitro-o-toluidine
GC-MS	EPA 8270C / 8270D	7,12-Dimethylbenz(a)anthracene
GC-MS	EPA 8270C / 8270D	Acetophenone
GC-MS	EPA 8270C / 8270D	Aramite
GC-MS	EPA 8270C / 8270D	Atrazine
GC-MS	EPA 8270C / 8270D	Biphenyl
GC-MS	EPA 8270C / 8270D	Chlorobenzilate
GC-MS	EPA 8270C / 8270D	Diallate
GC-MS	EPA 8270C / 8270D	Dibenzo(a,j)acridine
GC-MS	EPA 8270C / 8270D	Dimethoate
GC-MS	EPA 8270C / 8270D	Dinoseb
GC-MS	EPA 8270C / 8270D	Diphenyl ether
GC-MS	EPA 8270C / 8270D	Disulfoton
GC-MS	EPA 8270C / 8270D	Ethyl methacrylate
GC-MS	EPA 8270C / 8270D	Ethyl methanesulfonate
GC-MS	EPA 8270C / 8270D	Ethyl parathion
GC-MS	EPA 8270C / 8270D	Famphur
GC-MS	EPA 8270C / 8270D	Hexachlorophene
GC-MS	EPA 8270C / 8270D	Hexachloropropene
GC-MS	EPA 8270C / 8270D	Isodrin
GC-MS	EPA 8270C / 8270D	Isosafrole
GC-MS	EPA 8270C / 8270D	kepone
GC-MS	EPA 8270C / 8270D	Methapyrilene
GC-MS	EPA 8270C / 8270D	Methyl methanesulfonate
GC-MS	EPA 8270C / 8270D	Methyl parathion
GC-MS	EPA 8270C / 8270D	N-nitrosodiethylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosodi-n-butylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosomethylethylamine
GC-MS	EPA 8270C / 8270D	N-Nitrosomorpholine
GC-MS	EPA 8270C / 8270D	N-Nitrosopiperdine
GC-MS	EPA 8270C / 8270D	N-Nitrosopyrrolidine
GC-MS	EPA 8270C / 8270D	O,O,O-triethyl phosphorothi
GC-MS	EPA 8270C / 8270D	o-toluidine
GC-MS	EPA 8270C / 8270D	p-Dimethylaminoazobenze
GC-MS	EPA 8270C / 8270D	Pentachlorobenzene
GC-MS	EPA 8270C / 8270D	Pentachloroethane
GC-MS	EPA 8270C / 8270D	Pentachloronitrobenzene
GC-MS	EPA 8270C / 8270D	Phenacetin
GC-MS	EPA 8270C / 8270D	Phorate
GC-MS	EPA 8270C / 8270D	p-phenylenediamine
GC-MS	EPA 8270C / 8270D	Pronamide

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D	Safrole
GC-MS	EPA 8270C / 8270D	Sulfotepp
GC-MS	EPA 8270C / 8270D	Thionazin
GC-MS	EPA 8270C / 8270D SIM	Acenaphthene
GC-MS	EPA 8270C / 8270D SIM	Acenaphthylene
GC-MS	EPA 8270C / 8270D SIM	Anthracene
GC-MS	EPA 8270C / 8270D SIM	Azobenzene
GC-MS	EPA 8270C / 8270D SIM	Benzo(a)anthracene
GC-MS	EPA 8270C / 8270D SIM	benzo(a)pyrene
GC-MS	EPA 8270C / 8270D SIM	Benzo(b)fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Benzo(e)pyrene
GC-MS	EPA 8270C / 8270D SIM	Benzo(g,h,i)perylene
GC-MS	EPA 8270C / 8270D SIM	Benzo(k)fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Biphenyl
GC-MS	EPA 8270C / 8270D SIM	bis(2-chloroethyl)ether
GC-MS	EPA 8270C / 8270D SIM	bis(2-Ethylhexyl)phthalate
GC-MS	EPA 8270C / 8270D SIM	Carbazole
GC-MS	EPA 8270C / 8270D SIM	4-Chloro-3-methylphenol
GC-MS	EPA 8270C / 8270D SIM	2-Chlorophenol
GC-MS	EPA 8270C / 8270D SIM	Chrysene
GC-MS	EPA 8270C / 8270D SIM	Dibenzo(a,h)anthracene
GC-MS	EPA 8270C / 8270D SIM	2,4-Dichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,6-Dimethylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	2,4-Dimethylphenol
GC-MS	EPA 8270C / 8270D SIM	Fluoranthene
GC-MS	EPA 8270C / 8270D SIM	Fluorene
GC-MS	EPA 8270C / 8270D SIM	Hexachlorobenzene
GC-MS	EPA 8270C / 8270D SIM	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 8270C / 8270D SIM	1-Methylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	2-Methylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	1-Methylphenanthrene
GC-MS	EPA 8270C / 8270D SIM	Naphthalene
GC-MS	EPA 8270C / 8270D SIM	n-Nitrosodimethylamine
GC-MS	EPA 8270C / 8270D SIM	n-Nitroso-di-n-propylamine
GC-MS	EPA 8270C / 8270D SIM	Pentachlorophenol
GC-MS	EPA 8270C / 8270D SIM	Perylene
GC-MS	EPA 8270C / 8270D SIM	Phenanthrene
GC-MS	EPA 8270C / 8270D SIM	Phenol
GC-MS	EPA 8270C / 8270D SIM	Pyrene
GC-MS	EPA 8270C / 8270D SIM	2,4,5-Trichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,4,6-Trichlorophenol
GC-MS	EPA 8270C / 8270D SIM	2,3,5-Trimethylnaphthalene
GC-MS	EPA 8270C / 8270D SIM	1,4-Dioxane

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	EPA 8270C / 8270D SIM	Butylbenzylphthalate
GC-MS	EPA 8270C / 8270D SIM	Diethylphthalate
GC-MS	EPA 8270C / 8270D SIM	Dimethylphthalate
GC-MS	EPA 8270C / 8270D SIM	Di-n-butylphthalate
GC-MS	EPA 8270C / 8270D SIM	Di-n-octylphthalate
HPLC	EPA 8310	Acenaphthene
HPLC	EPA 8310	Acenaphthylene
HPLC	EPA 8310	Anthracene
HPLC	EPA 8310	Benzo(a)anthracene
HPLC	EPA 8310	Benzo(a)pyrene
HPLC	EPA 8310	Benzo(b)fluoranthene
HPLC	EPA 8310	Benzo(g,h,i)perylene
HPLC	EPA 8310	Benzo(k)fluoranthene
HPLC	EPA 8310	Chrysene
HPLC	EPA 8310	Dibenzo(a,h)anthracene
HPLC	EPA 8310	Fluoranthene
HPLC	EPA 8310	Fluorene
HPLC	EPA 8310	Indeno(1,2,3-cd)pyrene
HPLC	EPA 8310	1-Methylnaphthalene
HPLC	EPA 8310	2-Methylnaphthalene
HPLC	EPA 8310	Naphthalene
HPLC	EPA 8310	Phenanthrene
HPLC	EPA 8310	Pyrene
HPLC	EPA 8330A	HMX
HPLC	EPA 8330A	RDX
HPLC	EPA 8330A	1,3,5-TNB
HPLC	EPA 8330A	1,3-DNB
HPLC	EPA 8330A	Tetryl
HPLC	EPA 8330A	Nitrobenzene
HPLC	EPA 8330A	2,4,6-TNT
HPLC	EPA 8330A	4-AM-2,6-DNT
HPLC	EPA 8330A	2-AM-4,6-DNT
HPLC	EPA 8330A	2,6-DNT
HPLC	EPA 8330A	2,4-DNT
HPLC	EPA 8330A	2-Nitrotoluene
HPLC	EPA 8330A	4-Nitrotoluene
HPLC	EPA 8330A	3-Nitrotoluene
HPLC	EPA 8330A	3,5-Dinitroaniline
HPLC	EPA 8330A	2,4-Diamino-6-nitrotoluene
HPLC	EPA 8330A	2,6-Diamino-4-nitrotoluene
HPLC	EPA 8330A	Picric Acid

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC	EPA 8332	Nitroglycerine
HPLC	EPA 8332	PETN
Combustion-IR	EPA 9060A	TOC
IC	EPA 9056 / 9056A	Bromate
IC	EPA 9056 / 9056A	Bromide
IC	EPA 9056 / 9056A	Chloride
IC	EPA 9056 / 9056A	Fluoride
IC	EPA 9056 / 9056A	Nitrate
IC	EPA 9056 / 9056A	Nitrite
IC	EPA 9056 / 9056A	Phosphate
IC	EPA 9056 / 9056A	Sulfate
IC	EPA 9056 / 9056A	Chlorate
GC	EPA 8151A	Acifluorfen
GC	EPA 8151A	Bentazon
GC	EPA 8151A	Chloramben
GC	EPA 8151A	2,4-D
GC	EPA 8151A	2,4-DB
GC	EPA 8151A	Dacthal
GC	EPA 8151A	Dalapon
GC	EPA 8151A	Dicamba
GC	EPA 8151A	3,5-Dichlorobenzoic acid
GC	EPA 8151A	Dichlorprop
GC	EPA 8151A	Dinoseb
GC	EPA 8151A	MCPA
GC	EPA 8151A	MCPP
GC	EPA 8151A	Pentachlorophenol
GC	EPA 8151A	Picloram
GC	EPA 8151A	Silvex
GC	EPA 8151A	2,4,5-T
Spectrometric	EPA 9014	Cyanide
Gravimetric	EPA 9071B	Oil & Grease
GFAA	CA 939M	Organo Lead
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Purge & Trap	EPA 5030B / EPA 5035	Volatiles Prep
Acid Digestion	EPA 3050B	Metals Prep
Alkaline Digestion	EPA 3060A	Hexavalent Chrom
Soxhlet	EPA 3540C	Organic Extraction
Sonication	EPA 3550C	Organic Extraction
Waste Dilution	EPA 3580A	Organic Extraction
Microwave	EPA 3546	Organic Extraction
TCLP	EPA 1311	Leaching

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
SPLP	EPA 1312	Leaching
Florecil Clean-up	EPA 3620C	Extract Clean-Up
GPC Clean-up	EPA 3640A	Extract Clean-Up
Sulfur Clean-up	EPA 3660B	Extract Clean-Up
Acid/Permanganate Clean-up	EPA 3665A	Extract Clean-Up

<b>Air and Emissions</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	TO-15	1,1,1-trichloroethane
GC-MS	TO-15	1,1,2,2-tetrachloroethane
GC-MS	TO-15	1,1,2-Trichloro1,2,2-trifluoroethane
GC-MS	TO-15	1,1,2-trichloroethane
GC-MS	TO-15	1,1-dichloroethane
GC-MS	TO-15	1,1-Dichloroethene
GC-MS	TO-15	1,2,4-trichlorobenzene
GC-MS	TO-15	1,2,4-trimethylbenzene
GC-MS	TO-15	1,2-dibromoethane
GC-MS	TO-15	1,2-dichlorobenzene
GC-MS	TO-15	1,2-dichloroethane
GC-MS	TO-15	1,2-dichloroethene
GC-MS	TO-15	1,2-dichloropropane
GC-MS	TO-15	1,3,5-trimethylbenzene
GC-MS	TO-15	1,3-Butadiene
GC-MS	TO-15	1,3-Butadiene, 1,1,2,3,4,Hexachloro
GC-MS	TO-15	1,3-dichlorobenzene
GC-MS	TO-15	1,4-dichlorobenzene
GC-MS	TO-15	1,4-Dioxane
GC-MS	TO-15	2,2,4-Trimethylpentane
GC-MS	TO-15	4-Ethyltoluene
GC-MS	TO-15	Acetone
GC-MS	TO-15	Acrylonitrile
GC-MS	TO-15	Allyl Chloride
GC-MS	TO-15	Benzene
GC-MS	TO-15	Benzyl Chloride
GC-MS	TO-15	Bromodichloromethane
GC-MS	TO-15	Bromoform
GC-MS	TO-15	Bromomethane
GC-MS	TO-15	Carbon Disulfide
GC-MS	TO-15	Carbon Tetrachloride

<b>Air and Emissions</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC-MS	TO-15	Chlorobenzene
GC-MS	TO-15	Chloroethane
GC-MS	TO-15	Chloroethene
GC-MS	TO-15	Chloroform
GC-MS	TO-15	Chloromethane
GC-MS	TO-15	cis-1,3-Dichloropropene
GC-MS	TO-15	Cyclohexane
GC-MS	TO-15	Dibromochloromethane
GC-MS	TO-15	Dichlorodifluoromethane
GC-MS	TO-15	Dichlorotetrafluoroethane
GC-MS	TO-15	Ethyl Acetate
GC-MS	TO-15	Ethylbenzene
GC-MS	TO-15	Isopropyl Alcohol
GC-MS	TO-15	m+p-Xylene
GC-MS	TO-15	Methyl butyl Ketone
GC-MS	TO-15	Methyl Ethyl Ketone
GC-MS	TO-15	Methyl Isobutyl Ketone
GC-MS	TO-15	Methyl Tert-Butyl Ether
GC-MS	TO-15	Methylene Chloride
GC-MS	TO-15	n-Heptane
GC-MS	TO-15	n-Hexane
GC-MS	TO-15	o-Xylene
GC-MS	TO-15	Styrene
GC-MS	TO-15	Tetrachloroethylene
GC-MS	TO-15	Tetrahydrofuran
GC-MS	TO-15	Toluene
GC-MS	TO-15	Trans-1,2-Dichloroethene
GC-MS	TO-15	trans-1,3-Dichloropropene
GC-MS	TO-15	Trichloroethylene
GC-MS	TO-15	Trichloromonofluoromethan
GC-MS	TO-15	Vinyl Acetate
GC-MS	TO-15	Vinyl Bromide

**Notes:**

- 1) This laboratory offers commercial testing service.

Approved by:   
**R. Douglas Leonard**  
 Chief Technical Officer

Date: September 25, 2014



STATE WATER RESOURCES CONTROL BOARD  
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE



ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

## CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

**EMAX Laboratories, Inc.**

1835 West 205th Street

Torrance, CA 90501

Scope of the certificate is limited to the  
"Fields of Testing"  
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection,  
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of  
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2672**

Expiration Date: **6/30/2017**

Effective Date: **7/1/2015**

A handwritten signature in black ink, appearing to read "Christine Sotelo".

Sacramento, California  
subject to forfeiture or revocation

Christine Sotelo, Chief  
Environmental Laboratory Accreditation Program



**CALIFORNIA STATE  
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM  
Accredited Fields of Testing**



**EMAX Laboratories, Inc.**

1835 West 205th Street  
Torrance, CA 90501  
Phone: (310) 618-8889

**Certificate No.: 2672  
Renew Date: 6/30/2017**

**Field of Testing: 102 - Inorganic Chemistry of Drinking Water**

102.015	001	Hydrogen Ion (pH)	EPA 150.1
102.020	001	Turbidity	EPA 180.1
102.026	001	Calcium	EPA 200.7
102.026	002	Magnesium	EPA 200.7
102.026	003	Potassium	EPA 200.7
102.026	005	Sodium	EPA 200.7
102.026	006	Hardness (calculation)	EPA 200.7
102.030	001	Bromide	EPA 300.0
102.030	002	Chlorate	EPA 300.0
102.030	003	Chloride	EPA 300.0
102.030	005	Fluoride	EPA 300.0
102.030	006	Nitrate	EPA 300.0
102.030	007	Nitrite	EPA 300.0
102.030	008	Phosphate, Ortho	EPA 300.0
102.030	009	Sulfate	EPA 300.0
102.045	001	Perchlorate	EPA 314.0
102.095	001	Turbidity	SM2130B-2001
102.100	001	Alkalinity	SM2320B-1997
102.120	001	Hardness (calculation)	SM2340B-1997
102.121	001	Hardness	SM2340C-1997
102.130	001	Conductivity	SM2510B-1997
102.140	001	Residue, Filterable TDS	SM2540C-1997
102.150	001	Chloride	SM4110B
102.150	002	Fluoride	SM4110B
102.150	003	Nitrate	SM4110B
102.150	004	Nitrite	SM4110B
102.150	005	Phosphate, Ortho	SM4110B
102.150	006	Sulfate	SM4110B
102.170	001	Chloride	SM4500-CI- B-1997
102.190	001	Cyanide, Total	SM4500-CN E
102.192	001	Cyanide, amenable	SM4500-CN G
102.200	001	Fluoride	SM4500-F B,C-1997
102.203	001	Hydrogen Ion (pH)	SM4500-H+ B-2000
102.220	001	Nitrite	SM4500-NO2- B-2000
102.232	001	Nitrite	SM4500-NO3- E-2000
102.232	002	Nitrate	SM4500-NO3- E-2000
102.240	001	Phosphate, Ortho	SM4500-P E
102.260	001	Total Organic Carbon TOC	SM5310B

As of 6/30/2015, this list supersedes all previous lists for this certificate number.  
Customers: Please verify the current accreditation standing with the State.

102.270 001 Surfactants SM5540C

**Field of Testing: 103 - Toxic Chemical Elements of Drinking Water**

103.130 001	Aluminum	EPA 200.7
103.130 003	Barium	EPA 200.7
103.130 004	Beryllium	EPA 200.7
103.130 005	Cadmium	EPA 200.7
103.130 007	Chromium	EPA 200.7
103.130 008	Copper	EPA 200.7
103.130 009	Iron	EPA 200.7
103.130 011	Manganese	EPA 200.7
103.130 012	Nickel	EPA 200.7
103.130 015	Silver	EPA 200.7
103.130 017	Zinc	EPA 200.7
103.130 018	Boron	EPA 200.7
103.140 001	Aluminum	EPA 200.8
103.140 002	Antimony	EPA 200.8
103.140 003	Arsenic	EPA 200.8
103.140 004	Barium	EPA 200.8
103.140 005	Beryllium	EPA 200.8
103.140 006	Cadmium	EPA 200.8
103.140 007	Chromium	EPA 200.8
103.140 008	Copper	EPA 200.8
103.140 009	Lead	EPA 200.8
103.140 010	Manganese	EPA 200.8
103.140 012	Nickel	EPA 200.8
103.140 013	Selenium	EPA 200.8
103.140 014	Silver	EPA 200.8
103.140 015	Thallium	EPA 200.8
103.140 016	Zinc	EPA 200.8
103.140 017	Boron	EPA 200.8
103.140 018	Vanadium	EPA 200.8
103.160 001	Mercury	EPA 245.1
103.310 001	Chromium (VI)	EPA 218.6

**Field of Testing: 104 - Volatile Organic Chemistry of Drinking Water**

104.030 001	1,2-Dibromoethane	EPA 504.1
104.030 002	1,2-Dibromo-3-chloropropane	EPA 504.1
104.040 000	Volatile Organic Compounds	EPA 524.2
104.040 001	Benzene	EPA 524.2
104.040 007	n-Butylbenzene	EPA 524.2
104.040 008	sec-Butylbenzene	EPA 524.2
104.040 009	tert-Butylbenzene	EPA 524.2
104.040 010	Carbon Tetrachloride	EPA 524.2
104.040 011	Chlorobenzene	EPA 524.2
104.040 015	2-Chlorotoluene	EPA 524.2
104.040 016	4-Chlorotoluene	EPA 524.2
104.040 019	1,3-Dichlorobenzene	EPA 524.2

104.040	020	1,2-Dichlorobenzene	EPA 524.2
104.040	021	1,4-Dichlorobenzene	EPA 524.2
104.040	022	Dichlorodifluoromethane	EPA 524.2
104.040	023	1,1-Dichloroethane	EPA 524.2
104.040	024	1,2-Dichloroethane	EPA 524.2
104.040	025	1,1-Dichloroethene	EPA 524.2
104.040	026	cis-1,2-Dichloroethene	EPA 524.2
104.040	027	trans-1,2-Dichloroethene	EPA 524.2
104.040	028	Dichloromethane	EPA 524.2
104.040	029	1,2-Dichloropropane	EPA 524.2
104.040	033	cis-1,3-Dichloropropene	EPA 524.2
104.040	034	trans-1,3-Dichloropropene	EPA 524.2
104.040	035	Ethylbenzene	EPA 524.2
104.040	037	Isopropylbenzene	EPA 524.2
104.040	039	Naphthalene	EPA 524.2
104.040	041	N-propylbenzene	EPA 524.2
104.040	042	Styrene	EPA 524.2
104.040	043	1,1,1,2-Tetrachloroethane	EPA 524.2
104.040	044	1,1,2,2-Tetrachloroethane	EPA 524.2
104.040	045	Tetrachloroethene	EPA 524.2
104.040	046	Toluene	EPA 524.2
104.040	047	1,2,3-Trichlorobenzene	EPA 524.2
104.040	048	1,2,4-Trichlorobenzene	EPA 524.2
104.040	049	1,1,1-Trichloroethane	EPA 524.2
104.040	050	1,1,2-Trichloroethane	EPA 524.2
104.040	051	Trichloroethene	EPA 524.2
104.040	052	Trichlorofluoromethane	EPA 524.2
104.040	054	1,2,4-Trimethylbenzene	EPA 524.2
104.040	055	1,3,5-Trimethylbenzene	EPA 524.2
104.040	056	Vinyl Chloride	EPA 524.2
104.040	057	Xylenes, Total	EPA 524.2
104.040	061	Carbon Disulfide	EPA 524.2
104.040	062	Methyl Isobutyl Ketone	EPA 524.2
104.045	000	Trihalomethanes, Total	EPA 524.2
104.045	001	Bromodichloromethane	EPA 524.2
104.045	002	Bromoform	EPA 524.2
104.045	003	Chloroform	EPA 524.2
104.045	004	Dibromochloromethane	EPA 524.2
104.050	000	Gasoline Additives	EPA 524.2
104.050	002	Methyl tert-butyl Ether (MTBE)	EPA 524.2
104.050	003	tert-Amyl Methyl Ether (TAME)	EPA 524.2
104.050	004	Ethyl tert-butyl Ether (ETBE)	EPA 524.2
104.050	005	Trichlorotrifluoroethane	EPA 524.2
104.050	006	tert-Butyl Alcohol (TBA)	EPA 524.2

**Field of Testing: 108 - Inorganic Chemistry of Wastewater**

108.020	001	Conductivity	EPA 120.1
108.110	001	Turbidity	EPA 180.1

108.112	001	Boron	EPA 200.7
108.112	002	Calcium	EPA 200.7
108.112	003	Hardness (calculation)	EPA 200.7
108.112	004	Magnesium	EPA 200.7
108.112	005	Potassium	EPA 200.7
108.112	007	Sodium	EPA 200.7
108.113	001	Boron	EPA 200.8
108.113	002	Calcium	EPA 200.8
108.113	003	Magnesium	EPA 200.8
108.113	004	Potassium	EPA 200.8
108.113	006	Sodium	EPA 200.8
108.120	001	Bromide	EPA 300.0
108.120	002	Chloride	EPA 300.0
108.120	003	Fluoride	EPA 300.0
108.120	008	Sulfate	EPA 300.0
108.120	012	Nitrate (as N)	EPA 300.0
108.120	013	Nitrate-Nitrite (as N)	EPA 300.0
108.120	014	Nitrite as N	EPA 300.0
108.120	015	Phosphate, Ortho (as P)	EPA 300.0
108.220	002	Nitrite as N	EPA 352.1
108.323	001	Chemical Oxygen Demand	EPA 410.4
108.360	001	Phenols, Total	EPA 420.1
108.381	001	Oil and Grease	EPA 1664A
108.381	002	Oil & Grease Total	EPA 1664 Rev. B
108.385	001	Color	SM2120B-2001
108.390	001	Turbidity	SM2130B-2001
108.400	001	Acidity	SM2310B-1997
108.410	001	Alkalinity	SM2320B-1997
108.420	001	Hardness (calculation)	SM2340B-1997
108.421	001	Hardness	SM2340C-1997
108.430	001	Conductivity	SM2510B-1997
108.440	001	Residue, Total	SM2540B-1997
108.441	001	Residue, Filterable TDS	SM2540C-1997
108.442	001	Residue, Non-filterable TSS	SM2540D-1997
108.443	001	Residue, Settleable	SM2540F-1997
108.448	001	Bromide	SM4110B
108.448	002	Chloride	SM4110B
108.448	003	Fluoride	SM4110B
108.448	004	Nitrate	SM4110B
108.448	005	Nitrite	SM4110B
108.448	006	Nitrate-nitrite	SM4110B
108.448	007	Phosphate, Ortho	SM4110B
108.448	008	Sulfate	SM4110B
108.450	001	Chloride	SM4500-ChlorideB-1997
108.460	001	Chlorine, Total	SM4500-Cl B-2000
108.472	001	Cyanide, Total	SM4500-CN C,E-1999
108.473	001	Cyanide, amenable	SM4500-CN G-1999

108.480	001	Fluoride	SM4500-F B,C-1997
108.490	001	Hydrogen Ion (pH)	SM4500-H+ B-2000
108.504	002	Ammonia (as N)	SM4500-NH3 F-1997
108.505	002	Kjeldahl Nitrogen, Total (as N)	SM4500-NH3 F-1997
108.512	001	Kjeldahl Nitrogen, Total (as N)	SM4500-Norg C-1997
108.514	001	Nitrite as N	SM4500-NO2- B-2000
108.528	001	Nitrate-Nitrite (as N)	SM4500-NO3- E-2000
108.528	002	Nitrite as N	SM4500-NO3- E-2000
108.528	003	Nitrate (as N)	SM4500-NO3- E-2000
108.540	001	Phosphate, Ortho	SM4500-P E-1999
108.541	001	Phosphorus, Total	SM4500-P E-1999
108.552	001	Silica, Dissolved	SM4500-SiO2 C-1997
108.584	001	Sulfide (as S)	SM4500-S= D-2000
108.585	001	Sulfide (as S)	SM4500-S= F-2000
108.592	001	Biochemical Oxygen Demand	SM5210B-2001
108.595	001	Chemical Oxygen Demand	SM5220D-1997
108.596	001	Organic Carbon-Total (TOC)	SM5310B-2000
108.603	001	Oil & Grease Total	SM5520B-2001
108.605	001	Surfactants	SM5540C-2000
108.926	001	Cyanide, Total	Quickchem 10-204-00-1-X

**Field of Testing: 109 - Toxic Chemical Elements of Wastewater**

109.010	001	Aluminum	EPA 200.7
109.010	002	Antimony	EPA 200.7
109.010	003	Arsenic	EPA 200.7
109.010	004	Barium	EPA 200.7
109.010	005	Beryllium	EPA 200.7
109.010	006	Boron	EPA 200.7
109.010	007	Cadmium	EPA 200.7
109.010	009	Chromium	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	011	Copper	EPA 200.7
109.010	012	Iron	EPA 200.7
109.010	013	Lead	EPA 200.7
109.010	015	Manganese	EPA 200.7
109.010	016	Molybdenum	EPA 200.7
109.010	017	Nickel	EPA 200.7
109.010	019	Selenium	EPA 200.7
109.010	021	Silver	EPA 200.7
109.010	023	Thallium	EPA 200.7
109.010	024	Tin	EPA 200.7
109.010	025	Titanium	EPA 200.7
109.010	026	Vanadium	EPA 200.7
109.010	027	Zinc	EPA 200.7
109.020	001	Aluminum	EPA 200.8
109.020	002	Antimony	EPA 200.8
109.020	003	Arsenic	EPA 200.8
109.020	004	Barium	EPA 200.8

109.020	005	Beryllium	EPA 200.8
109.020	006	Cadmium	EPA 200.8
109.020	007	Chromium	EPA 200.8
109.020	008	Cobalt	EPA 200.8
109.020	009	Copper	EPA 200.8
109.020	010	Lead	EPA 200.8
109.020	011	Manganese	EPA 200.8
109.020	012	Molybdenum	EPA 200.8
109.020	013	Nickel	EPA 200.8
109.020	014	Selenium	EPA 200.8
109.020	015	Silver	EPA 200.8
109.020	016	Thallium	EPA 200.8
109.020	017	Vanadium	EPA 200.8
109.020	018	Zinc	EPA 200.8
109.020	021	Iron	EPA 200.8
109.020	022	Tin	EPA 200.8
109.020	023	Titanium	EPA 200.8
109.104	001	Chromium (VI)	EPA 218.6
109.190	001	Mercury	EPA 245.1
109.449	001	Iron	SM3500-Fe B-1997

**Field of Testing: 110 - Volatile Organic Chemistry of Wastewater**

110.040	000	Purgeable Organic Compounds	EPA 624
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**Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater**

111.100	000	Acid/base/neutral Organic Compounds	EPA 625
111.170	000	Pesticides & PCBs	EPA 608

**Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste**

114.010	001	Antimony	EPA 6010B
114.010	002	Arsenic	EPA 6010B
114.010	003	Barium	EPA 6010B
114.010	004	Beryllium	EPA 6010B
114.010	005	Cadmium	EPA 6010B
114.010	006	Chromium	EPA 6010B
114.010	007	Cobalt	EPA 6010B
114.010	008	Copper	EPA 6010B
114.010	009	Lead	EPA 6010B
114.010	010	Molybdenum	EPA 6010B
114.010	011	Nickel	EPA 6010B
114.010	012	Selenium	EPA 6010B
114.010	013	Silver	EPA 6010B
114.010	014	Thallium	EPA 6010B
114.010	015	Vanadium	EPA 6010B
114.010	016	Zinc	EPA 6010B
114.020	001	Antimony	EPA 6020
114.020	002	Arsenic	EPA 6020
114.020	003	Barium	EPA 6020
114.020	004	Beryllium	EPA 6020

114.020 005	Cadmium	EPA 6020
114.020 006	Chromium	EPA 6020
114.020 007	Cobalt	EPA 6020
114.020 008	Copper	EPA 6020
114.020 009	Lead	EPA 6020
114.020 010	Molybdenum	EPA 6020
114.020 011	Nickel	EPA 6020
114.020 012	Selenium	EPA 6020
114.020 013	Silver	EPA 6020
114.020 014	Thallium	EPA 6020
114.020 015	Vanadium	EPA 6020
114.020 016	Zinc	EPA 6020
114.103 001	Chromium (VI)	EPA 7196A
114.106 001	Chromium (VI)	EPA 7199
114.140 001	Mercury	EPA 7470A
114.141 001	Mercury	EPA 7471A
114.222 001	Cyanide	EPA 9014
114.230 001	Sulfides, Total	EPA 9034
114.240 001	Corrosivity - pH Determination	EPA 9040B
114.241 001	Corrosivity - pH Determination	EPA 9045C
114.250 001	Fluoride	EPA 9056
114.270 001	Fluoride	EPA 9214
114.280 001	Organic Lead	HML 939-M
114.280 001	Organic Lead	HML 939-M

**Field of Testing: 115 - Extraction Test of Hazardous Waste**

115.020 001	Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311
115.021 001	TCLP Inorganics	EPA 1311
115.022 001	TCLP Extractables	EPA 1311
115.023 001	TCLP Volatiles	EPA 1311
115.030 001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II
115.040 001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312

**Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste**

116.010 000	EDB and DBCP	EPA 8011
116.020 030	Nonhalogenated Volatiles	EPA 8015B
116.020 031	Ethanol and Methanol	EPA 8015B
116.030 001	Gasoline-range Organics	EPA 8015B
116.080 000	Volatile Organic Compounds	EPA 8260B
116.080 120	Oxygenates	EPA 8260B
116.110 001	Total Petroleum Hydrocarbons - Gasoline	LUFT

**Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste**

117.010 001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B
117.016 001	Diesel-range Total Petroleum Hydrocarbons	LUFT
117.110 000	Extractable Organics	EPA 8270C
117.140 000	Polynuclear Aromatic Hydrocarbons	EPA 8310
117.170 000	Nitroaromatics and Nitramines	EPA 8330
117.171 000	Nitroaromatics and Nitramines	EPA 8330A

117.210	000	Organochlorine Pesticides	EPA 8081A
117.220	000	PCBs	EPA 8082
117.240	000	Organophosphorus Pesticides	EPA 8141A
117.250	000	Chlorinated Herbicides	EPA 8151A

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**Field of Testing:** 120 - Physical Properties of Hazardous Waste

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120.010	001	Ignitability	EPA 1010
120.040	001	Reactive Cyanide	Section 7.3 SW-846
120.050	001	Reactive Sulfide	Section 7.3 SW-846
120.070	001	Corrosivity - pH Determination	EPA 9040B
120.080	001	Corrosivity - pH Determination	EPA 9045C

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CALIFORNIA  
**Water Boards**

STATE WATER RESOURCES CONTROL BOARD  
REGIONAL WATER QUALITY CONTROL BOARDS



CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

**CERTIFICATE OF ENVIRONMENTAL ACCREDITATION**

Is hereby granted to

**ALS Environmental - Houston HRMS**

10450 Stancliff Road, Suite 210

Houston, TX 77099

Scope of the certificate is limited to the  
"Fields of Testing"  
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection,  
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of  
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2452**

Expiration Date: **2/28/2017**

Effective Date: **3/1/2015**

Sacramento, California  
subject to forfeiture or revocation

Christine Sotelo, Chief  
Environmental Laboratory Accreditation Program



CALIFORNIA STATE  
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM  
Accredited Fields of Testing



ALS Environmental - Houston HRMS

10450 Stancliff Road, Suite 210  
Houston, TX 77099  
Phone: (713) 266-1599

Certificate No.: 2452  
Renew Date: 2/28/2017

**Field of Testing: 105 - Semi-volatile Organic Chemistry of Drinking Water**

105.230 000	Dioxins	EPA 1613
105.230 001	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	EPA 1613
105.230 002	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) Screening O	EPA 1613

**Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater**

111.111 000	Dioxins and Dibenzofurans	EPA 1613B
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**Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste**

117.120 000	Dioxins and Dibenzofurans	EPA 8280A
117.130 000	Dioxins and Dibenzofurans	EPA 8290

As of 6/23/2015, this list supersedes all previous lists for this certificate number.  
Customers: Please verify the current accreditation standing with the State.



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

ALS ENVIRONMENTAL – HOUSTON HRMS
19408 Park Row, Suite 320
Houston, Texas 77084
Rebecca Pierrot Phone: (713) 266 1599
rebecca.pierrot@alsglobal.com

ENVIRONMENTAL

Valid To: November 30, 2015

Certificate Number: 2897.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

High Resolution Gas Chromatography/High Resolution Mass Spectrometry

Table with 5 columns: Parameter/Analyte, Hazardous Waste (Non-Potable Water, Solid Hazardous Waste), Tissue, and Air/Emissions. Rows list PCB 1 through PCB 5 with corresponding EPA method numbers (1668A, B, C) for each category.

Peter Meyer (handwritten signature)

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 6	EPA 1668A EPA 1668B EPA 1668C			
PCB 7	EPA 1668A EPA 1668B EPA 1668C			
PCB 8	EPA 1668A EPA 1668B EPA 1668C			
PCB 9	EPA 1668A EPA 1668B EPA 1668C			
PCB 10	EPA 1668A EPA 1668B EPA 1668C			
PCB 11	EPA 1668A EPA 1668B EPA 1668C			
PCB 12	EPA 1668A EPA 1668B EPA 1668C			
PCB 13	EPA 1668A EPA 1668B EPA 1668C			
PCB 14	EPA 1668A EPA 1668B EPA 1668C			
PCB 15	EPA 1668A EPA 1668B EPA 1668C			
PCB 16	EPA 1668A EPA 1668B EPA 1668C			
PCB 17	EPA 1668A EPA 1668B EPA 1668C			
PCB 19	EPA 1668A EPA 1668B EPA 1668C			
PCB 20	EPA 1668A EPA 1668B EPA 1668C			
PCB 21	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 22	EPA 1668A EPA 1668B EPA 1668C			
PCB 23	EPA 1668A EPA 1668B EPA 1668C			
PCB 24	EPA 1668A EPA 1668B EPA 1668C			
PCB 25	EPA 1668A EPA 1668B EPA 1668C			
PCB 26	EPA 1668A EPA 1668B EPA 1668C			
PCB 27	EPA 1668A EPA 1668B EPA 1668C			
PCB 28	EPA 1668A EPA 1668B EPA 1668C			
PCB 29	EPA 1668A EPA 1668B EPA 1668C			
PCB 30	EPA 1668A EPA 1668B EPA 1668C			
PCB 31	EPA 1668A EPA 1668B EPA 1668C			
PCB 32	EPA 1668A EPA 1668B EPA 1668C			
PCB 33	EPA 1668A EPA 1668B EPA 1668C			
PCB 34	EPA 1668A EPA 1668B EPA 1668C			
PCB 35	EPA 1668A EPA 1668B EPA 1668C			
PCB 36	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 37	EPA 1668A EPA 1668B EPA 1668C			
PCB 38	EPA 1668A EPA 1668B EPA 1668C			
PCB 39	EPA 1668A EPA 1668B EPA 1668C			
PCB 40	EPA 1668A EPA 1668B EPA 1668C			
PCB 41	EPA 1668A EPA 1668B EPA 1668C			
PCB 42	EPA 1668A EPA 1668B EPA 1668C			
PCB 43	EPA 1668A EPA 1668B EPA 1668C			
PCB 44	EPA 1668A EPA 1668B EPA 1668C			
PCB 45	EPA 1668A EPA 1668B EPA 1668C			
PCB 46	EPA 1668A EPA 1668B EPA 1668C			
PCB 47	EPA 1668A EPA 1668B EPA 1668C			
PCB 48	EPA 1668A EPA 1668B EPA 1668C			
PCB 49	EPA 1668A EPA 1668B EPA 1668C			
PCB 50	EPA 1668A EPA 1668B EPA 1668C			
PCB 51	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 52	EPA 1668A EPA 1668B EPA 1668C			
PCB 53	EPA 1668A EPA 1668B EPA 1668C			
PCB 54	EPA 1668A EPA 1668B EPA 1668C			
PCB 55	EPA 1668A EPA 1668B EPA 1668C			
PCB 56	EPA 1668A EPA 1668B EPA 1668C			
PCB 57	EPA 1668A EPA 1668B EPA 1668C			
PCB 58	EPA 1668A EPA 1668B EPA 1668C			
PCB 59	EPA 1668A EPA 1668B EPA 1668C			
PCB 60	EPA 1668A EPA 1668B EPA 1668C			
PCB 61	EPA 1668A EPA 1668B EPA 1668C			
PCB 62	EPA 1668A EPA 1668B EPA 1668C			
PCB 63	EPA 1668A EPA 1668B EPA 1668C			
PCB 64	EPA 1668A EPA 1668B EPA 1668C			
PCB 65	EPA 1668A EPA 1668B EPA 1668C			
PCB 66	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 67	EPA 1668A EPA 1668B EPA 1668C			
PCB 68	EPA 1668A EPA 1668B EPA 1668C			
PCB 69	EPA 1668A EPA 1668B EPA 1668C			
PCB 70	EPA 1668A EPA 1668B EPA 1668C			
PCB 71	EPA 1668A EPA 1668B EPA 1668C			
PCB 72	EPA 1668A EPA 1668B EPA 1668C			
PCB 73	EPA 1668A EPA 1668B EPA 1668C			
PCB 74	EPA 1668A EPA 1668B EPA 1668C			
PCB 75	EPA 1668A EPA 1668B EPA 1668C			
PCB 76	EPA 1668A EPA 1668B EPA 1668C			
PCB 77	EPA 1668A EPA 1668B EPA 1668C			
PCB 78	EPA 1668A EPA 1668B EPA 1668C			
PCB 79	EPA 1668A EPA 1668B EPA 1668C			
PCB 80	EPA 1668A EPA 1668B EPA 1668C			
PCB 81	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 82	EPA 1668A EPA 1668B EPA 1668C			
PCB 83	EPA 1668A EPA 1668B EPA 1668C			
PCB 84	EPA 1668A EPA 1668B EPA 1668C			
PCB 85	EPA 1668A EPA 1668B EPA 1668C			
PCB 86	EPA 1668A EPA 1668B EPA 1668C			
PCB 87	EPA 1668A EPA 1668B EPA 1668C			
PCB 88	EPA 1668A EPA 1668B EPA 1668C			
PCB 89	EPA 1668A EPA 1668B EPA 1668C			
PCB 90	EPA 1668A EPA 1668B EPA 1668C			
PCB 91	EPA 1668A EPA 1668B EPA 1668C			
PCB 92	EPA 1668A EPA 1668B EPA 1668C			
PCB 93	EPA 1668A EPA 1668B EPA 1668C			
PCB 94	EPA 1668A EPA 1668B EPA 1668C			
PCB 95	EPA 1668A EPA 1668B EPA 1668C			
PCB 96	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 97	EPA 1668A EPA 1668B EPA 1668C			
PCB 98	EPA 1668A EPA 1668B EPA 1668C			
PCB 99	EPA 1668A EPA 1668B EPA 1668C			
PCB 100	EPA 1668A EPA 1668B EPA 1668C			
PCB 101	EPA 1668A EPA 1668B EPA 1668C			
PCB 102	EPA 1668A EPA 1668B EPA 1668C			
PCB 103	EPA 1668A EPA 1668B EPA 1668C			
PCB 104	EPA 1668A EPA 1668B EPA 1668C			
PCB 105	EPA 1668A EPA 1668B EPA 1668C			
PCB 106	EPA 1668A EPA 1668B EPA 1668C			
PCB 107	EPA 1668A EPA 1668B EPA 1668C			
PCB 108	EPA 1668A EPA 1668B EPA 1668C			
PCB 109	EPA 1668A EPA 1668B EPA 1668C			
PCB 110	EPA 1668A EPA 1668B EPA 1668C			
PCB 111	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 112	EPA 1668A EPA 1668B EPA 1668C			
PCB 113	EPA 1668A EPA 1668B EPA 1668C			
PCB 114	EPA 1668A EPA 1668B EPA 1668C			
PCB 115	EPA 1668A EPA 1668B EPA 1668C			
PCB 116	EPA 1668A EPA 1668B EPA 1668C			
PCB 117	EPA 1668A EPA 1668B EPA 1668C			
PCB 118	EPA 1668A EPA 1668B EPA 1668C			
PCB 119	EPA 1668A EPA 1668B EPA 1668C			
PCB 120	EPA 1668A EPA 1668B EPA 1668C			
PCB 121	EPA 1668A EPA 1668B EPA 1668C			
PCB 122	EPA 1668A EPA 1668B EPA 1668C			
PCB 123	EPA 1668A EPA 1668B EPA 1668C			
PCB 124	EPA 1668A EPA 1668B EPA 1668C			
PCB 125	EPA 1668A EPA 1668B EPA 1668C			
PCB 126	EPA 1668A EPA 1668B EPA 1668C			

*Peter Abney*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 127	EPA 1668A EPA 1668B EPA 1668C			
PCB 128	EPA 1668A EPA 1668B EPA 1668C			
PCB 129	EPA 1668A EPA 1668B EPA 1668C			
PCB 130	EPA 1668A EPA 1668B EPA 1668C			
PCB 131	EPA 1668A EPA 1668B EPA 1668C			
PCB 132	EPA 1668A EPA 1668B EPA 1668C			
PCB 133	EPA 1668A EPA 1668B EPA 1668C			
PCB 134	EPA 1668A EPA 1668B EPA 1668C			
PCB 135	EPA 1668A EPA 1668B EPA 1668C			
PCB 136	EPA 1668A EPA 1668B EPA 1668C			
PCB 137	EPA 1668A EPA 1668B EPA 1668C			
PCB 138	EPA 1668A EPA 1668B EPA 1668C			
PCB 139	EPA 1668A EPA 1668B EPA 1668C			
PCB 140	EPA 1668A EPA 1668B EPA 1668C			
PCB 141	EPA 1668A EPA 1668B EPA 1668C			

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 142	EPA 1668A EPA 1668B EPA 1668C			
PCB 143	EPA 1668A EPA 1668B EPA 1668C			
PCB 144	EPA 1668A EPA 1668B EPA 1668C			
PCB 145	EPA 1668A EPA 1668B EPA 1668C			
PCB 146	EPA 1668A EPA 1668B EPA 1668C			
PCB 147	EPA 1668A EPA 1668B EPA 1668C			
PCB 148	EPA 1668A EPA 1668B EPA 1668C			
PCB 149	EPA 1668A EPA 1668B EPA 1668C			
PCB 150	EPA 1668A EPA 1668B EPA 1668C			
PCB 151	EPA 1668A EPA 1668B EPA 1668C			
PCB 152	EPA 1668A EPA 1668B EPA 1668C			
PCB 153	EPA 1668A EPA 1668B EPA 1668C			
PCB 154	EPA 1668A EPA 1668B EPA 1668C			
PCB 155	EPA 1668A EPA 1668B EPA 1668C			
PCB 156	EPA 1668A EPA 1668B EPA 1668C			

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 157	EPA 1668A EPA 1668B EPA 1668C			
PCB 158	EPA 1668A EPA 1668B EPA 1668C			
PCB 159	EPA 1668A EPA 1668B EPA 1668C			
PCB 160	EPA 1668A EPA 1668B EPA 1668C			
PCB 161	EPA 1668A EPA 1668B EPA 1668C			
PCB 162	EPA 1668A EPA 1668B EPA 1668C			
PCB 163	EPA 1668A EPA 1668B EPA 1668C			
PCB 164	EPA 1668A EPA 1668B EPA 1668C			
PCB 165	EPA 1668A EPA 1668B EPA 1668C			
PCB 166	EPA 1668A EPA 1668B EPA 1668C			
PCB 167	EPA 1668A EPA 1668B EPA 1668C			
PCB 168	EPA 1668A EPA 1668B EPA 1668C			
PCB 169	EPA 1668A EPA 1668B EPA 1668C			
PCB 170	EPA 1668A EPA 1668B EPA 1668C			
PCB 171	EPA 1668A EPA 1668B EPA 1668C			

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 172	EPA 1668A EPA 1668B EPA 1668C			
PCB 173	EPA 1668A EPA 1668B EPA 1668C			
PCB 174	EPA 1668A EPA 1668B EPA 1668C			
PCB 175	EPA 1668A EPA 1668B EPA 1668C			
PCB 176	EPA 1668A EPA 1668B EPA 1668C			
PCB 177	EPA 1668A EPA 1668B EPA 1668C			
PCB 178	EPA 1668A EPA 1668B EPA 1668C			
PCB 179	EPA 1668A EPA 1668B EPA 1668C			
PCB 180	EPA 1668A EPA 1668B EPA 1668C			
PCB 181	EPA 1668A EPA 1668B EPA 1668C			
PCB 182	EPA 1668A EPA 1668B EPA 1668C			
PCB 183	EPA 1668A EPA 1668B EPA 1668C			
PCB 184	EPA 1668A EPA 1668B EPA 1668C			
PCB 185	EPA 1668A EPA 1668B EPA 1668C			
PCB 186	EPA 1668A EPA 1668B EPA 1668C			

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 187	EPA 1668A EPA 1668B EPA 1668C			
PCB 188	EPA 1668A EPA 1668B EPA 1668C			
PCB 189	EPA 1668A EPA 1668B EPA 1668C			
PCB 190	EPA 1668A EPA 1668B EPA 1668C			
PCB 191	EPA 1668A EPA 1668B EPA 1668C			
PCB 192	EPA 1668A EPA 1668B EPA 1668C			
PCB 193	EPA 1668A EPA 1668B EPA 1668C			
PCB 194	EPA 1668A EPA 1668B EPA 1668C			
PCB 195	EPA 1668A EPA 1668B EPA 1668C			
PCB 196	EPA 1668A EPA 1668B EPA 1668C			
PCB 197	EPA 1668A EPA 1668B EPA 1668C			
PCB 198	EPA 1668A EPA 1668B EPA 1668C			
PCB 199	EPA 1668A EPA 1668B EPA 1668C			
PCB 200	EPA 1668A EPA 1668B EPA 1668C			
PCB 201	EPA 1668A EPA 1668B EPA 1668C			

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
PCB 202	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 203	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 204	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 205	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 206	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 207	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 208	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
PCB 209	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C	EPA 1668A EPA 1668B EPA 1668C
Sample Preparation	Liquid Liquid Extraction	Liquid Extraction, Soxhlet	Soxhlet	Soxhlet
<b>Dioxins/Furans</b>				
2,3,7,8-TCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,7,8-PeCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,4,7,8-HxCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,6,7,8-HxCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23

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Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
1,2,3,7,8,9-HxCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,4,6,7,8-HpCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
OCDD	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
2,3,7,8-TCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,7,8-PeCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
2,3,4,7,8-PeCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,4,7,8-HxCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,6,7,8-HxCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,7,8,9-HxCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
2,3,4,6,7,8-HxCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
1,2,3,4,6,7,8-HpCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23

*Peter Almyer*

Parameter/Analyte	Hazardous Waste		Tissue	Air/Emissions
	Non-Potable Water	Solid Hazardous Waste		
1,2,3,4,7,8,9-HpCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
OCDF	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	EPA 1613B EPA 8290 EPA 8290A EPA 8280A	TO-9A M23
Sample Preparation	Liquid-Liquid Extraction	Liquid Extraction , Soxhlet	Soxhlet	Soxhlet

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below, and for the test methods applicable to ISO IEC 17025:2005:

Parameter/Analyte	Potable Water	Hazardous Waste		Foods
		Non-Potable Water	Solid	
<b>Dioxins/Furans</b>				
2,3,7,8-TCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8-PeCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,6,7,8-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8,9-HxCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,6,7,8-HpCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
OCDD	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
2,3,7,8-TCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8-PeCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
2,3,4,7,8-PeCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,6,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,7,8,9-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
2,3,4,6,7,8-HxCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,6,7,8-HpCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
1,2,3,4,7,8,9-HpCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
OCDF	EPA 1613B	EPA 1613B	EPA 1613B	EPA 1613B
Sample Preparation	Liquid/Liquid	Liquid/liquid	Soxhlet	Soxhlet,

*Peter Almyer*



American Association for Laboratory Accreditation

# *Accredited DoD ELAP Laboratory*

A2LA has accredited

## **ALS ENVIRONMENTAL HOUSTON HRMS**

*Houston, TX*

for technical competence in the field of

### **Environmental Testing**

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality System Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Presented this 28<sup>th</sup> day of January 2014.





President & CEO  
For the Accreditation Council  
Certificate Number 2897.01  
Valid to November 30, 2015

*For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.*

## **Appendix B**

# **Biological Avoidance and Minimization Plan**

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Draft

**Biological Avoidance and Minimization Plan**  
**Remedial Investigation at**  
**Installation Restoration Program Site 6**

**Defense Fuel Support Point San Pedro,**  
**San Pedro, California**

Contract Number: N62473-09-D-2622

Contract Task Order: 0100

Document Control Number: KCH-2622-0100-0009

August 2015

Prepared for



**Department of the Navy**  
**Naval Facilities Engineering Command**  
**Southwest**

Prepared by



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# Acronyms and Abbreviations

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BAMP	biological avoidance and minimization plan
bgs	below ground surface
CAGN	coastal California gnatcatcher
CDFW	California Department of Fish and Wildlife
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COPCs	chemicals of potential concern
DFSP	Defense Fuel Support Point
DOT	United States Department of Transportation
DWR	California Department of Water Resources
ESA	Endangered Species Act
FS	feasibility study
HSA	hollow-stem auger
IRP	Installation Restoration Program
JP-4	jet propulsion fuel, type 4
KCH	CH2M HILL Kleinfelder, a Joint Venture
NAVFAC	Naval Facilities Engineering Command
Navy	United States Department of the Navy
OSWER	Office of Solid Waste and Emergency Response
PAH	polycyclic aromatic hydrocarbon
PVB	Palos Verdes blue butterfly
RI	remedial investigation
SI	site investigation
SVOC	semivolatile organic compound
TPH	total petroleum hydrocarbon
TriEco-Tt	TriEco-Tetra Tech
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service

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# 1.0 Introduction

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CH2M HILL Kleinfelder, A Joint Venture (KCH), has prepared this biological avoidance and minimization plan (BAMP) to perform a remedial investigation (RI) at Installation Restoration Program (IRP) Site 6 at Defense Fuel Support Point (DFSP) San Pedro, San Pedro, California (Figure 1-1). KCH is performing this work for the United States Department of the Navy (Navy), Naval Facilities Engineering Command (NAVFAC) Southwest in accordance with Contract No. N62473-09-D-2622, Contract Task Order No. 0100.

IRP Site 6 is a former disposal area referred to as the South Ravine (Figure 1-2). RI activities will include sampling of soil and groundwater.

This BAMP has been prepared for the regulatory agencies, including the United States Fish and Wildlife Service (USFWS), to address the potential presence of species protected under the federal Endangered Species Act (ESA) – specifically, the threatened coastal California gnatcatcher (*Polioptila californica californica*; CAGN) and the endangered Palos Verdes blue butterfly (*Glaucopsyche lygdamus palosverdesensis*; PVB). It is intended that this BAMP will provide the information necessary to coordinate with USFWS in the manner described in the United States Environmental Protection Agency's (USEPA's) Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Compliance with Other Laws Manual (Office of Solid Waste and Emergency Response [OSWER] Directive 9234.1-02) (1989). This BAMP describes the following:

- Natural resources that could be affected by the proposed fieldwork
- How field activities could affect federally listed species
- Actions that will be taken to avoid or minimize impacts to federally listed species

## 1.1 IRP Site 6 Description

IRP Site 6 was defined after a 1990 site visit identified the South Ravine as a former disposal area. Paint spills, rusted 55-gallon drums, and 1- and 5-gallon cans containing varying amounts of unidentified liquids were noted. Wooden boards and furniture, brush, metal pipe, concrete, and tires were also visible during the site visit.

The northern portion of the South Ravine has northwest and northeast branches (Figure 1-3). The northwest branch has been filled almost to grade. During the site inspection, a fill depth of 19 feet below ground surface (bgs) was identified in this branch. The northeast branch was identified as being incised (narrower and with steeper sides), with fill visible in this area as well (Jacobs, 1993).

Engineering of the south-sloping ravine bottom in IRP Site 6 is apparent in a 1943 topographic map (United States Navy Fuel Depot, 1943). A concrete v-drain was present during the 1990 site visit in the south end of the ravine bottom. Sands present in this area may be from the upper part of the ravine where vertically cut banks are visible.

The history of disposal in the ravine is unknown. Base personnel did not know of filling operations, so it is likely that disposal at this site was unscheduled (Jacobs, 1993).

## 1.2 Summary of Previous Investigations

A fuel spill occurred in 1981 or 1982 at a nearby tank (Figure 1-2). An estimated 10,000 gallons of diesel fuel reportedly flowed east into the southern end of the South Ravine. No cleanup of the spill was attempted. Two borings were placed in the probable path of the spill by Woodward-Clyde Consultants. Soil samples collected from 2 to 15 feet bgs from WCB-10 and 2 to 4 feet bgs from WCB-11 were analyzed for total petroleum hydrocarbons (TPHs) as gasoline, kerosene, diesel, jet propulsion fuel, type 4 (JP-4), and unknown hydrocarbons. There were no detections above the detection limit of 10 milligrams per kilogram for the four samples submitted (Jacobs, 1993).

A site investigation (SI) was conducted at IRP Site 6 in 1992. IRP Site 6 consists of a main north-south trending drainage with a small southeast trending tributary ravine (northwest branch of the South Ravine; Figure 1-3). During the site inspection, along the surface of the main drainage, miscellaneous debris, including metal, glass, and wood fragments were present. The upper portions of the tributary drainage were covered with asphalt-like substance, possibly the remnants of a fuel spill from Tank 5 (Jacobs, 1993).

Six soil borings (B01 through B06) were installed during the SI. During the boring installations, Jacobs (1993) noted that the main ravine and tributary were filled with approximately 10 to 20 feet of assorted fill debris. Abundant broken glass fragments were encountered at boring B01 in the northern end of the ravine. At boring B05, volatile organic compounds (benzene, ethylbenzene, toluene, xylenes, and various ketones) and semivolatile organic compounds (SVOCs; primarily polycyclic aromatic hydrocarbons [PAHs]) were detected in soil. Although the soil was logged as fill material to a depth of 14 feet bgs, the lower portions of this interval may be alluvium with abundant residual fuel products. The soils were overlaid with silty soil fill materials.

The main ravine axis was also capped with a granular fill sand that was apparently emplaced during installation of a concrete v-drain. The v-drain is confined to the main ravine axis. At the south end of the ravine, rip-rap is present along the v-drain in the vicinity of boring B06. No fill soils were encountered in B06. Boring B07 was drilled east of the northern part of IRP Site 6. Clean sands of the San Pedro Formation were encountered in this boring (Jacobs, 1993).

Sampling at IRP Site 6 during the SI indicated that elevated levels of chemicals of potential concern (COPCs) were present, particularly in the northwestern portion of the ravine. COPCs identified during the SI include fuels, organic lead, metals, SVOCs, and organochlorine pesticides. An RI/feasibility study (FS) was recommended for IRP Site 6 based on these exceedances.

## 2.0 Proposed Action: Remedial Investigation

---

The RI at IRP Site 6 will be implemented using the approaches outlined in the following subsections. Field sampling will last approximately 6 weeks and is scheduled for October 2015.

### 2.1 Utility Locating/Geophysical Survey

A geophysical survey will be conducted at IRP Site 6 to assess the lateral extent of landfill debris to aid in the placement of sampling locations. This effort is necessary in the northern half of the site where debris is expected. Due to thick vegetation and steep terrain in the northeastern ravine, a traditional geophysical grid is not practical. Instead, geophysical transect lines will be used. The lines will consist of approximately 4,000 feet of linear coverage with one transect parallel to each main ravine and several transects perpendicular across the ravines. Actual transect locations will be identified in the field, based on access considerations.

The geophysical subcontractor will also clear subsurface utilities at IRP Site 6 sample locations before drilling and trenching activities begin. All clearances needed for borehole drilling will be obtained in accordance with the Navy's established procedures and requirements. All locations will be marked in the field with a wooden stake, and the sample location will be indicated on a map.

### 2.2 Soil Sampling

Soil samples will be collected via hand augering, HSA drilling, and trenching.

#### 2.2.1 Hand Augering

A hand auger will be used to clear each boring location down to approximately 5 feet bgs at IRP Site 6. The hand auger will also be used to collect soil samples from five soil borings to 10 feet bgs (Figure 1-3).

#### 2.2.2 Hollow-Stem Auger Drilling

Prior to drilling at IRP Site 6, each boring location will be cleared down to approximately 5 feet bgs with a decontaminated hand auger. Seven borings will be advanced by hollow-stem auger (HSA) drilling using a track-mounted HSA drill rig equipped with 8-inch-diameter augers. The location of the borings are shown on Figure 1-3.

#### 2.2.3 Trenching

Twelve trenches will be excavated using an all-terrain backhoe with extendable bucket boom at IRP Site 6 (Figure 1-3). The trenches will be 2 to 3 feet wide, 10 feet long, and will extend to an estimated depth of 10 feet bgs (contingent upon soil conditions to keep an open, stable trench).

Trench locations were partially based on access requirements for the mechanized equipment. Actual trench locations will be based in part on the results of the geophysical survey, visual observations of potential waste or debris, and avoidance of sensitive biological resources to the extent feasible.

When sample collection is complete, the trench will be backfilled with the original material unless there are visible signs of contamination. Backfilled soil will be compacted with the backhoe bucket.

## **2.2.4 Soil Sampling**

Soil samples will be collected directly from the hand auger, using a California modified split-spoon sampler lined with stainless steel sleeves, or from the excavator bucket. Each hand auger borehole will be backfilled by pouring bentonite chips into the borehole to within a few inches of the surface and hydrating. Each HSA borehole will be backfilled with cement grout with 5 percent bentonite to within a few inches of the surface. All boreholes will be patched with native soil to match existing surface conditions.

## **2.3 Monitoring Well Installation**

Two monitoring wells will be installed at IRP Site 6 to evaluate whether COPCs in groundwater have been adequately characterized and whether COPCs pose an unacceptable risk to human receptors (Figure 1-3). Well boreholes will be advanced using HSA drilling. Monitoring wells will be installed and constructed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981; 1990).

## **2.4 Monitoring Well Development**

New monitoring wells MW01 and MW02 will be developed and existing monitoring wells SB-10 and B-21 will be redeveloped (Figure 1-3) prior to groundwater sampling. Monitoring well development will be performed consistent with *Bulletin 74-81* and *Bulletin 74-90: Water Well Standards: State of California* (DWR, 1981; 1991).

Development water will be transferred to United States Department of Transportation (DOT)-approved 55-gallon drums. The drums will be sealed, labeled, and stored in a secure location designated by the Navy.

## **2.5 Groundwater Sampling**

The goal of groundwater sampling at IRP Site 6 is to evaluate whether COPCs have been adequately characterized in groundwater at IRP Site 6 and whether they pose an unacceptable risk to human receptors. New monitoring wells (MW-01 and MW-02) and existing monitoring wells (SB-10 and B-21; Figure 1-3) will be monitored and sampled.

## 3.0 Existing Conditions, Including Federally Listed Species within the Action Area

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Vegetation at IRP Site 6 is anticipated to be similar to vegetation at IRP Site 32 that consists of chaparral and grassland species including mustard (*Hirschfeldia incana*), thistle (*Centaurea melitensis*), and various wild grasses such as wild oat (*Avena fatua*). Previous reports included inventories of plants and animals found in and around DFSP San Pedro (Jacobs, 1993; OHM Remedial Services Corp., 2001), but surveys specific to IRP Site 6 were not performed.

Potential bird species at IRP Site 6 include the common raven (*Corvus corax*), American kestrel (*Falco sparverius*), American robin (*Turdus migratorius*), California towhee (*Melospiza crissalis*), European starling (*Sturnus vulgaris*), house finch (*Carpodacus mexicanus*), mourning dove (*Zenaidura macroura*), rock pigeon (*Columba livia*), and western meadowlark (*Sturnella neglecta*).

Mammals potentially present at IRP Site 6 include species of small rodents and larger mammals. Rodents include the California ground squirrel (*Spermophilus beecheyi*), deer mouse (*Peromyscus maniculatus*), dusky-footed woodrat (*Neotoma fuscipes*), fox squirrel (*Sciurus niger*), and western harvest mouse (*Reithrodontomys megalotis*). Larger mammals include badgers (*Taxidea taxus*), black-tailed jackrabbits (*Lepus californicus*), coyote (*Canis latrans*), gray foxes (*Urocyon cinereoargenteus*), raccoons (*Procyon lotor*), red foxes (*Vulpes vulpes*), and striped skunks (*Mephitis mephitis*).

A number of special-status species could occur at IRP Site 6, including but not limited to the federally listed as threatened CAGN and the federally listed as endangered PVB (CDFW, 2014). No USFWS-designated critical habitat occurs on DFSP San Pedro (USFWS, 1984; 2014a).

The ecological setting information presented in this section was taken from the RI Report for IRP Site 32 (TriEco-Tt, 2013).

### 3.1 Coastal California Gnatcatcher

The CAGN was listed as threatened by USFWS in March 1993. This species is a non-migratory songbird that nests and forages in moderately dense stands of coastal sage scrub occurring on arid hillsides, mesas, and washes. The breeding season for CAGN is 15 February through 31 August (USFWS, 2008). Loss of suitable habitat and fragmentation of habitat from expanding development and agriculture have been a major factor in the decline of this species. The Palos Verdes Peninsula is considered a “core” population area, supporting 30 or more pairs of CAGN (Atwood et al., 1998; Mock, 2014). Based on information included in the California Department of Fish and Wildlife Natural Diversity Database, DFSP San Pedro was estimated to hold 10 percent of the Palos Verdes Peninsula population in 1993 (CDFW, 2014). In 2011, focused surveys to determine the presence or absence of CAGN were conducted at DFSP San Pedro (ICF International, 2011). The study

did not consider the proposed RI at IRP Site 6 as potentially suitable habitat for CAGN (ICF International, 2011). Although CAGN was observed at DFSP San Pedro consistently, the coastal sage scrub habitat at the southern end of DFSP San Pedro, was unoccupied and generally supported a lower diversity of coastal sage scrub species (ICF International, 2011).

## 3.2 Palos Verdes Blue Butterfly

The PVB, a subspecies of the silvery blue butterfly, was listed as endangered in 1980 (USFWS, 1984). The species was believed extinct for 11 years until it was discovered at DFSP San Pedro in March 1994 (Mattoni, 1994). In 1995, following the rediscovery of the species, a captive propagation effort was started along with efforts to restore habitat (Mattoni et al., 1998 [2003]).

The PVB is found in coastal sage scrub and is dependent on two known host plants, southern California milkvetch (*Astragalus trichopodus* var. *lonchus*) and common deerweed (*Acmispon glaber*), and completes its life cycle near its host plant. Eggs are typically laid in the flower heads of the host plant and larvae pupate in the leaf litter (Center for Biological Diversity, 2014). The adult flight period for PVB occurs in the spring. Although the adult flight period is reported to occur from late January through early May (USFWS, 2014b), at DFSP San Pedro, the adult flight period has been observed as typically occurring from late February through early May. At DFSP San Pedro, the first observation of PVB (in flight) occurred between 21 February and 16 March, and the last observation occurred between 26 March and 5 May over the course of 20 years (1994 to 2013) of annual population surveys (Longcore and Osborne, 2014). PVB appears to prefer early succession coastal sage scrub to later succession species (e.g., *Artemisia californica*, *Eriogonum fasciculatum*, and *Encelia californica*) replace host plants (Longcore and Osborne, 2011).

According to a 2013 survey, the geographic distribution of the butterfly (not counting release sites) is limited to the northern half of DFSP San Pedro (i.e., the current nursery and behind the old nursery) north of the South Ravine (Attachment 1; Longcore and Osborne 2011; 2014). Annual population surveys use a single transect with multiple segments. Transect segment 9, which surrounds the South Ravine, was added in 1996. PVB were recorded at transect segment 9 from 1997 to 2008 (excluding 1998 and 2001; Longcore and Osborne, 2011), but have not been observed at that location during the last five annual surveys (Longcore and Osborne, 2014).

The South Ravine was mapped as a potential area for habitat restoration when the species was rediscovered in 1994 (Mattoni, 1994). In the 2011 survey report, a pilot disturbance experiment that mechanically disturbed (scraped) 1 acre of habitat around transect segment 9 was underway (Longcore and Osborne, 2011). By 2012, significant quantities of young deerweed plants were present, but by 2013 PVB had neither colonized nor had been released at that location, although PVB releases may be permitted in the future (Longcore and Osborne, 2012; 2014). Further efforts to create early successional habitats for PVB at DFSP San Pedro are recommended through clearing of vegetation through targeted disturbance (Longcore and Osborne, 2011).

## 4.0 Avoidance/Minimization Measures

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The following measures are proposed to avoid and minimize potential effects to biological resources, including federally listed species, within the action area of the proposed action:

1. Sampling activities associated with the RI at IRP Site 6 will be kept to the smallest footprint feasible, while still meeting RI objectives.
2. A biological field monitor will be onsite during fieldwork. The biological field monitor will have the authority to halt field activities, if necessary, to avoid impacts to CAGN and/or PVB.
3. To the maximum extent practicable, fieldwork and other project-related field activities will take place from 1 September through 14 February, outside the CAGN breeding season (15 February through 31 August) and outside the typical DFSP San Pedro PVB adult flight period (late February through early May).
4. If avoiding CAGN breeding season is not practicable, then the following additional measures will be employed for CAGN before or during the breeding season:
  - a. Preconstruction surveys will be conducted by a USFWS 10(A)1(a) recovery permitted biologist for active nests within 500 feet of the proposed work area during the breeding season, at least 1 week before fieldwork.
  - b. If an active CAGN nest (nest containing eggs or nestlings) occurs within 500 feet of the proposed action area, the biological monitor will maintain a 500-foot buffer distance around the nest, with project activities directed to other areas, until the young fledge or at least 10 days after the nest has been identified as being abandoned – determined to have failed due to circumstances not related to project activities.
5. Trench locations will take into account avoidance of coastal sage scrub and especially milkvetch and common deerweed to the maximum extent feasible. All trenches will be filled the same day as excavation. No trenches will be left open. If unusual circumstances occur where a trench needs to be left open overnight, it will be covered to avoid wildlife entrapment. Topsoil will be set aside on clean plastic sheeting during excavation and used as final cover on backfill.
6. Workers will be instructed in southern California milkvetch and common deerweed identification by the onsite biological field monitor. Workers will access the sampling locations by a route that avoids southern milkvetch and deerweed to the maximum extent practicable. All vegetation clearing activities and locations will be coordinated with the ongoing PVB habitat restoration efforts at DFSP San Pedro to the maximum extent practicable to further efforts to create early successional habitats for PVB.
7. No night-time construction activities will be planned, and lights are not anticipated. If lighting is required for extraordinary circumstances (for example, completion of well construction in an unstable borehole), then lighting structures will be shielded so that

light is not directed towards plant communities occupied by CAGN or PVB. The biological monitor will have the authority to halt field activities if necessary to avoid impacting CAGN and PVB.

8. Workers will be prohibited from bringing dogs or domesticated pets to the site to ensure that domestic pets do not affect wildlife through harassment or predation in adjacent natural habitats.
9. Personnel may not purposely harm or kill wildlife.
10. All trash will be collected and removed from the site daily or temporarily stored onsite within a suitable container with a complete closure (lid).

## 5.0 Assessment of Potential Effects

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### 5.1 Coastal California Gnatcatcher

Potential direct effects to CAGN could include temporary disturbance such as noise and dust from RI field activities. These effects could occur during the approximate 1 month of field activities. No effect to breeding or nesting CAGN is anticipated due the proposed avoidance measures: avoiding the breeding season or maintaining a 500-foot buffer around nests. Vegetation at IRP Site 6 primarily consists of chaparral and grassland species, but coastal sage scrub will be avoided to the maximum extent feasible. Therefore, no CAGN habitat loss, temporary or permanent, is anticipated. In addition, no indirect effects to CAGN are anticipated. Therefore, the proposed action may affect, but is not likely to adversely affect, CAGN.

### 5.2 Palos Verdes Blue Butterfly

Potential direct effects to PVB could include temporary disturbance such as dust and ground vibration from RI field activities. Potential direct effects to PVB could also include the loss of habitat and host plants as well as the possibility of crushing PVB pupae in the leaf litter. RI field activities including soil borings, trenches, and hand augering are proposed within the South Ravine habitat restoration area, which contains young deerweed plants. The direct impacts to the habitat restoration area are anticipated to be approximately 1/8<sup>th</sup> of an acre. By keeping the disturbance footprint as small as feasible, avoiding the PVB flight period, and avoiding PVB host plants to the maximum extent feasible, direct effects to PVB are expected to be minimized.

Potential indirect effects could include increased human access to monitor well locations. However, wells will be accessible by foot, and vehicles will remain on existing dirt roads. Potential indirect effects could also include permanent modifications to habitat in the South Ravine. It is anticipated that by coordinating with PVB habitat restoration efforts at DFSP San Pedro on the selection of vegetation clearance and disturbance areas to the maximum extent practicable additional, successional habitats suitable for PVB will be created. Therefore, the proposed action may affect, but is not likely to adversely affect, PVB.

### 5.3 Conclusion

The proposed RI at IRP Site 6 may affect, but will not likely adversely affect, the threatened CAGN or the endangered PVB. This determination is based on the implementation of the avoidance and minimization measures, the temporary nature of the majority of the potential effects, the relatively small area of potential effect, and the relatively short duration of the activities.

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## 6.0 References

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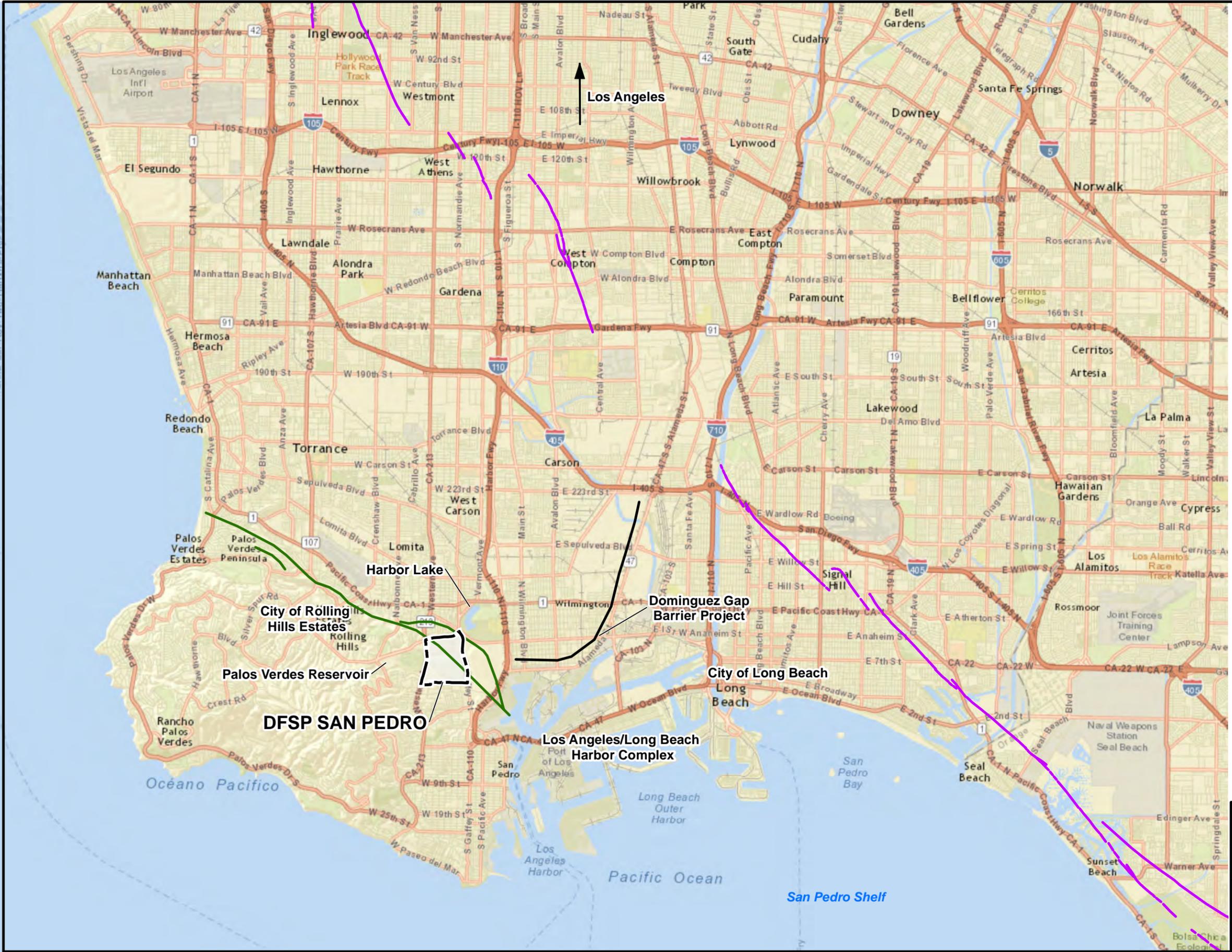
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## Figures

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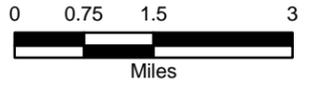
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- LEGEND**
-  NEWPORT-INGLEWOOD FAULT
  -  PALOS VERDES FAULT
  -  DFSP SAN PEDRO BOUNDARY

**NOTES:**  
 DFSP = Defense Fuel Support Point  
 RI = Remedial Investigation  
 IRP = Installation Restoration Program

**IMAGERY SOURCE:**  
 ESRI ArcGIS Online Web Service, Streets



**Site Vicinity**

Biological Avoidance and Minimization Plan, RI at IRP Site 6  
 Defense Fuel Support Point San Pedro, California

  
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FIGURE  
**1-1**

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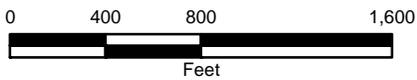
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**LEGEND**

-  IRP SITE BOUNDARY
-  FORMER NAVY HOUSING UNIT
-  DFSP SAN PEDRO BOUNDARY

NOTES:  
 DFSP = Defense Fuel Support Point  
 RI = Remedial Investigation  
 IRP = Installation Restoration Program



SOURCE:  
 ESRI ArcGIS Online Web Service,  
 World Imagery 5/5/2010

**IRP Site 6 Location**

Biological Avoidance and Minimization Plan, RI at IRP Site 6  
 Defense Fuel Support Point San Pedro, California



FIGURE

**1-2**

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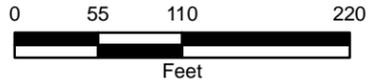
**LEGEND**

- EXISTING GROUNDWATER MONITORING WELL
- HISTORICAL SOIL BORING
- PROPOSED HAND AUGER BORING
- PROPOSED SOIL BORING BORING
- PROPOSED GROUNDWATER MONITORING WELL
- PROPOSED TRENCH
- IRP SITE 6 BOUNDARY

NOTES:  
 RI = Remedial Investigation  
 IRP = Installation Restoration Program

Approximate groundwater flow direction based on October 2013 groundwater elevation measurements (SGI, 2014).

IMAGERY SOURCE:  
 ESRI ArcGIS Online Web Service, World Imagery  
 5/25/2010



**IRP Site 6 Sample Locations**

Biological Avoidance and Minimization Plan, RI at IRP Site 6  
 Defense Fuel Support Point San Pedro, California

FIGURE  
**1-3**

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**Attachment 1**  
**Location of Palos Verdes Blue Butterfly Transect at DFSP and**  
**Palos Verdes Housing, Figure 2 (Longcore and Osborne, 2011)**

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**Figure 2.** Location of Palos Verdes blue butterfly transect at DFSP (segments 1–10) and Palos Verdes housing (segment 11), as found on the Torrance, California 7.5' USGS quadrangle.

### 2.3 Occupancy Analysis

We tested for trends in occupancy of Palos Verdes blue butterfly by constructing a multiple logistic regression, in which the independent continuous variables were year and estimated population size and the dependent categorical variable was presence or absence of butterflies along each transect segment. While the dependent variable may exhibit some degree of spatial autocorrelation, the well-documented asynchronous fluctuation of abundance among transect

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