SAP Worksheet #1: Title and Approval Page

Final

TIER II SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)

November 2016

Bioremediation Operation, Maintenance, and Reporting
Installation Restoration Site 70
Naval Weapons Station Seal Beach
Seal Beach, California

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EXECUTIVE SUMMARY

This sampling and analysis plan has been prepared to describe long-term monitoring activities associated with the enhanced in situ bioremediation (EISB) in accordance with the *Final Remedial Action Work Plan Implementation of Enhanced In Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (ECC-Geosyntec, 2008) for Installation Restoration (IR) Site 70, at Naval Weapons Station (NAVWPNSTA) Seal Beach, Seal Beach, California (Figure 1). This work is being performed for the Navy, Naval Facilities Engineering Command Southwest, under Contract No. N62473-12-D-2005, Contract Task Order 0016.

IR Site 70 was the former National Aeronautics and Space Administration Research Testing and Evaluation Area, located on the NAVWPNSTA Seal Beach property. NAVWPNSTA Seal Beach is an active federal facility. The lead agency for remedial action at this station is the U.S. Department of the Navy. Regulatory agencies providing support and oversight include the California Department of Toxic Substances Control and the California Regional Water Quality Control Board—Santa Ana Region.

IR Site 70 encompasses approximately 40 acres, the groundwater contains elevated concentrations of trichloroethene (TCE) and the plume extends beyond the site boundaries. The remedial action objective for IR Site 70 was to install an EISB system to treat the impacted groundwater and to reduce potential threats to human health and the surrounding environment. The EISB system was designed and implemented from 2007 through 2010 to facilitate a more rapid dechlorination of TCE to ethene. TCE degradation products such as 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, and vinyl chloride were anticipated to be formed. Chloroform, perchloroethylene, and 1,1-dichloroethane were identified as compounds of concern in the *Record of Decision / Remedial Action Plan (ROD/RAP), Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (Geosyntech, 2006a). Therefore, chloroform, 1,1-dichloroethane, TCE, perchloroethylene, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, and vinyl chloride have been identified as contaminants of concern for IR Site 70. Target cleanup goals have been established for the identified contaminants of concern and listed in Worksheet #10. Target cleanup goals are the California maximum contaminant levels for drinking water (Title 22 of the California Code of Regulations).

From 2007 through 2010, monitoring wells, injection wells, and compliance wells were installed as part of the EISB system (Figures 2 and 3). In 2008, groundwater was sampled to established baseline groundwater conditions. After baseline sampling event, amendments were injected into groundwater as part of EISB treatment. Supplemental amendments have been periodically

injected into the groundwater since the initial injection event. Since the baseline sampling event in 2008, groundwater has been sampled semiannually to monitor natural attenuation and the EISB progress. This sampling and analysis plan will describe the sampling and analytical requirements necessary to continue to monitor the EISB remediation progress at IR Site 70.

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List of Acronyms

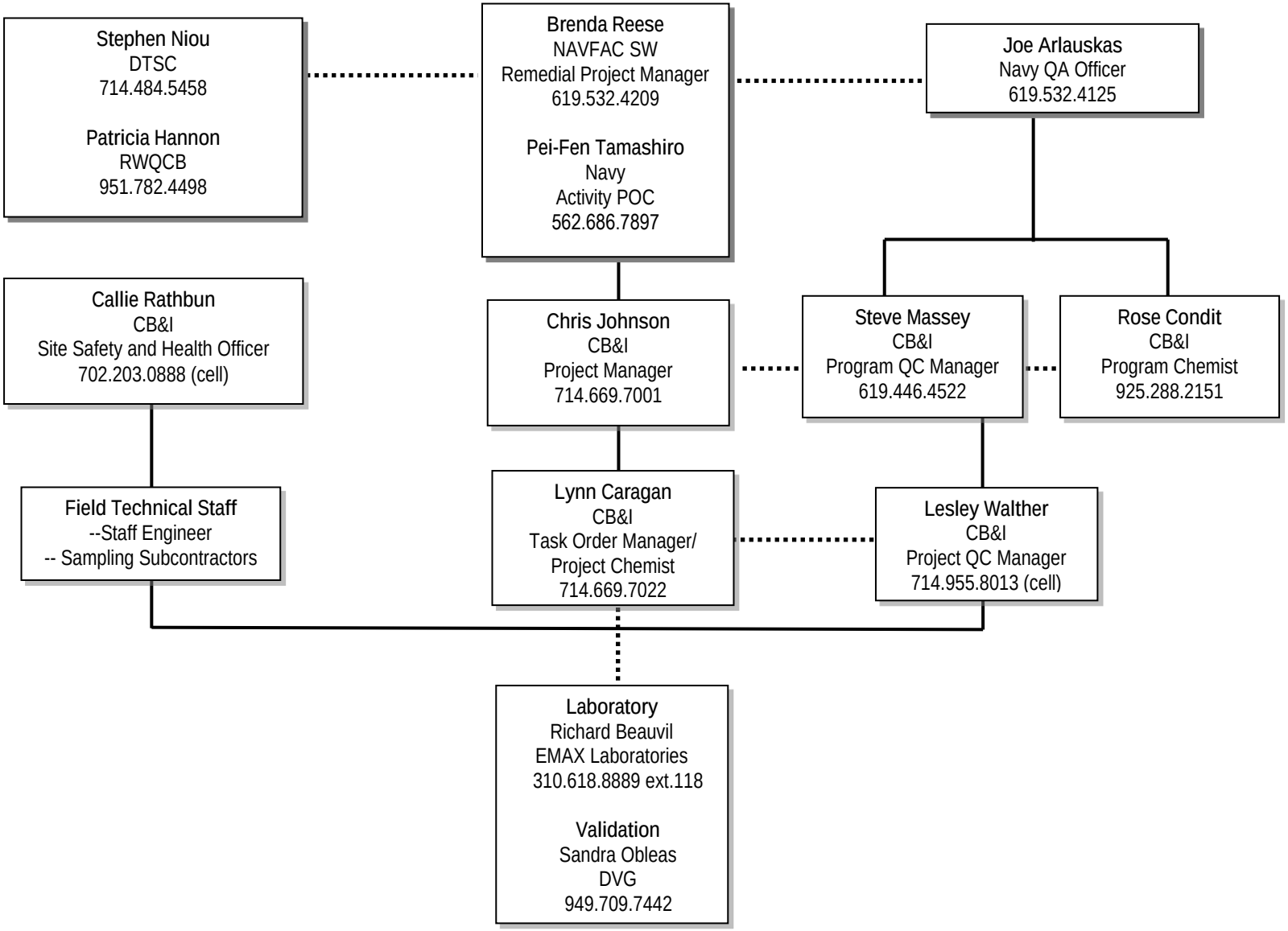
%	percent
<	less than
≤	less than or equal to
≥	greater than or equal to
°C	degree Celsius
µg/L	microgram per liter
bgs	below ground surface
CB&I	CB&I Federal Services LLC
COC	contaminant of concern
CSM	conceptual site model
DHC/vcrA	dehalococcoides gene/vinyl chloride reductase A gene
DHG	dissolved hydrogen gas
DO	dissolved oxygen
DoD	U.S. Department of Defense
EDD	electronic data deliverable
EISB	enhanced in situ bioremediation
EPA	U.S. Environmental Protection Agency
EVO	emulsified vegetable oil
H&S	health and safety
ICAL	initial calibration
IR	Installation Restoration
LCD	laboratory control duplicate
LCS	laboratory control sample
LOD	limit of detection
LOQ	limit of quantitation
MB	method blank
MCL	maximum contaminant level
mL	milliliter
MNA	monitor natural attenuation
MS	matrix spike
MSD	matrix spike duplicate
NAVFAC SW	Naval Facilities Engineering Command Southwest
NAVWPNSTA	Naval Weapons Station
Navy	U.S. Department of the Navy
ORP	oxidation-reduction potential
PCE	perchloroethylene
PM	project manager
POC	point of contact
QA	quality assurance
QAO	Quality Assurance Officer
QC	quality control
QCM	Quality Control Manager
QL	quantitation limit

List of Acronyms (continued)

QSM	*Quality System Manual for Environmental Laboratories, Version 5.0*
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
SAP	sampling and analysis plan
SOP	standard operating procedure
SSHO	site safety and health officer
TCE	trichloroethene
TCG	target cleanup goal
TDS	total dissolved solids
TO	total organics
TOC	total organic carbon
VC	vinyl chloride
VFA	volatile fatty acid
VOC	volatile organic compound

SAP Worksheet #2: Project Organizational Chart

All lines of responsibility (solid lines) and lines of communication (dotted lines) are provided.



SAP Worksheet #2: Project Organizational Chart (continued)

Notes:

CB&I	*CB&I Federal Services LLC*
DTSC	*California Department of Toxic Substances Control*
DVG	*Data Validation Group*
EPA	*U.S. Environmental Protection Agency*
NAVFAC SW	*Naval Facilities Engineering Command Southwest*
POC	*point of contact*
QA	*quality assurance*
QC	*quality control*
RWQCB	*California Regional Water Quality Control Board—Santa Ana Region*

SAP Worksheet #3: Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure
Communication with Regulators	Naval Facilities Engineering Command Southwest (NAVFAC SW) Remedial Project Manager (RPM) Naval Weapons Station (NAVWPNSTA) Seal Beach Activity Point of Contact (POC)	Brenda Reese Pei-Fen Tamashiro	619.532.4209 562.626.7897	The NAVFAC SW RPM and NAVWPNSTA Activity POC will be the primary POC for communication with regulators for project information. The NAVFAC SW RPM and the NAVWPNSTA Activity POC will communicate any project field changes, construction, or quality issues to the regulators.
POC with NAVFAC SW RPM	CB&I Federal Services LLC (CB&I) Project Manager (PM) CB&I Task Order Manager	Chris Johnson Lynn Caragan	714.669.7001 714.669.7022	All materials and information about the project will be forwarded to RPM by the PM and the Task Order Manager.
Sampling and Analysis Plan (SAP) Changes in the Field	Project Chemist Program Chemist	Lynn Caragan Rose Condit	714.669.7022 925.288.2151	The Project Chemist is responsible for documenting field changes related to sampling and for informing or seeking approval from the Program Chemist or NAVFAC SW Quality Assurance Officer (QAO). The Project Chemist or Program Chemist is also responsible for generating SAP amendments as necessary for approval by the NAVFAC SW QAO. The Project Chemist oversees the documentation, notification, and corrective actions associated with sampling issues in writing. Due to the potential impact field changes and SAP amendments may have on the project, the Project Chemist is to be notified of such issues within 24 hours or one business day.

SAP Worksheet #3: Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure
Stop Work Issues (Quality)	NAVFAC SW QAO	Joe Arlauskas	619.532.4125	The NAVFAC QAO is authorized to suspend project execution if quality assurance (QA) requirements are not adequately followed. The NAVFAC QAO will notify the NAVFAC SW RPM and CB&I Program Quality Control Manager if quality control (QC) issues requiring Stop Work are identified. The CB&I Program QC Manager or Program Chemist, in consultation with the PM and NAVFAC SW RPM, will work with the NAVFAC QAO to resolve QA issues and resume work. Upon resolution, the Project Quality Control Manager (QCM) oversees the documentation, notification, and corrective actions associated with site issues in writing within five business days.
Sampling Quality Issues	Project QCM Program QCM	Lesley Walther Steve Massey	714.955.8013 619.446.4522	In general, the Project Chemist is the POC for sampling and chemistry issues and the Project QCM is the POC for other quality issues. If quality issues are not resolved at the project level (in consultation with the Task Order Manager, or Technical Manager, etc.), then the issue will be elevated to the Program Chemist or QCM.
Health And Safety (H&S) Issues	Site Safety and Health Officer (SSHO)	Callie Rathbun	702.203.0888	The SSHO is the POC for H&S issues. If H&S issues are not resolved at the project level (in consultation with the PM, Site Supervisor, Technical Manager, etc.), then the issue will be elevated to the Program Health and Safety Officer. The Program Health and Safety Officer or designee will seek additional guidance or approval from the U.S. Department of the Navy (Navy) Health and Safety Officer, if necessary. Upon resolution, the SSHO oversees the documentation, notification, and corrective actions associated with the issue in writing. Due to the potential seriousness of H&S issues, the SSHO is to be notified of H&S issues immediately.

SAP Worksheet #3: Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure
Sample Collection Issues	Project Chemist Program Chemist	Lynn Caragan Rose Condit	714.669.7002 925.288.2151	The Project Chemist is the POC for sampling and chemistry issues. If sampling issues are not resolved at the project level (in consultation with the PM, Site Supervisor, Technical Manager, Geologist, etc.), then the issue will be elevated to the Program Chemist or QCM. The Program Chemist or QCM will seek additional guidance or approval from the NAVFAC QAO, if necessary. Upon resolution, the Project Chemist oversees the documentation, notification, and corrective actions associated with the QA issue in writing. Due to the potential impact sampling issues may have on the project, the Project Chemist is to be notified of sampling issues within 24 hours or one business day.
Amendments to Approved *Uniform Federal Policy for Quality Assurance Plans (UFP/QAPP)* (U.S. Environmental Protection Agency [EPA], 2005)	Project Chemist Program Chemist	Lynn Caragan Rose Condit	714.669.7022 925.288.2151	If field activities change to such a degree the original exiting SAP does not adequately cover the work being performed, then a SAP addendum will be written. SAP addendum will follow the requirements of *Environmental Work Instruction EVR.2—Review, Approval, Revision, and Amendment of Sampling and Analysis Plans (SAPs)* (NAVFAC SW, 2011). SAP addendums may be written by the Project Chemist or the Program Chemist. SAP addendums will be reviewed and approved by the NAVFAC QAO prior to regulatory review or field implementation.
Stop Work Issues (H&S)	SSHO	Callie Rathbun	702.203.0888	All employees have the right and duty to Stop Work when conditions are unsafe, or when established safety procedures are being disregarded. Whenever an employee determines that workplace conditions present an immediate uncontrolled risk of injury or illness, immediate resolution with the appropriate supervisor shall be sought. Should the supervisor be unable or unwilling to correct the unsafe conditions, the employee is authorized and required to issue a Stop Work Order in accordance with CB&I procedure CMS-710-05-PR-00400, “Stop Work Authority.” The specific activity or operation in question shall be discontinued until the issue is resolved.

SAP Worksheet #3: Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Telephone Number	Procedure
Laboratory Reporting or Data Quality Issues	Project Chemist Program Chemist	Lynn Caragan Rose Condit	714.669.7022 925.288.2151	The Project Chemist is the POC for laboratory issues. The project data manager is the POC for electronic data deliverables (EDDs). If laboratory issues are not resolved with the Project Chemist or data manager, then the laboratory issue will be elevated to the Program Chemist. Upon resolution, the Project Chemist oversees the documentation, notification, and corrective actions associated with the laboratory issue in writing.
Notification of Non-usable Analytical Data	Program Chemist	Rose Condit	925.288.2151	If significant problems are identified by the laboratory or the project team that impact the usability of the data (e.g., the data is rejected or the data quality objectives are not met), the Program Chemist will notify the CB&I PM and the NAVFAC SW RPM and the NAVWPNSTA Seal Beach POC within 24 hours or the next business day. CB&I will also notify the NAVFAC SW QAO if directed by the NAVFAC SW PRM.

SAP Worksheet #4: Project Scoping Session Participants Sheet

Project Name: Bioremediation Operation, Maintenance, and Reporting at IR Site 70	Site Name: IR Site 70
Projected Date(s) of Sampling: December 2016 and 2017; July 2017 and 2018	Site Location: NAVWPNSTA Seal Beach, CA
Project Manager: Chris Johnson	

Date of Session: August 9, 0216

Scoping Session Purpose: Project Kickoff Meeting

Name	Title	Affiliation	Phone #	Email Address	Project Role
Brenda Reese	NAVFAC SW RPM	NAVFAC SW	619.532.4590	brenda.reese@navy.mil	RPM
Pei-Fen Tamashiro	NAVWPNSTA Seal Beach POC	Navy	562.626.7897	pei-fen.tamashiro@navy.mil	NAVWPNSTA Seal Beach POC
Chris Johnson	PM	CB&I	714.669.7001	chris.e.johnson@cbifederalservices.com	CB&I PM
Lynn Caragan	Task Order Manager	CB&I	714.669.7022	lynn.caragan@cbifederalservices.com	CB&I Task Order Manager
Lesley Walther	Project QC Manager	CB&I	714.955.8013	lesley.walther@cbifederalservices.com	CB&I Project QC Manager

Comments/Decisions:

Project description, organization and staffing, schedule, logistics, security, and site access

Action Items:

CB&I to prepare a Tier II sampling analysis plan, accident prevention plan addendum, second five-year review plan report.

Consensus Decisions:

- Groundwater monitoring events to occur in December 2016, July 2017, December 2017, and July 2018.
- Volatile organic compounds (VOCs) analysis in soil gas is not needed at every vapor probes. CB&I to recommend number of probes to sample to generate a representative data set.
- Accident prevention plan addendum to be reviewed by the NAVWPNSTA Seal Beach.

SAP Worksheet #5: Conceptual Site Model

The conceptual site model (CSM) for Installation Restoration (IR) Site 70 was first presented in Section 2.4 of the *Final Remedial Action Work Plan Implementation of Enhanced In Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (ECC-Geosyntec, 2008). The CSM has since been updated based on data presented in the *Draft 2015 Annual Performance Monitoring Report, Enhanced In Situ bioremediation Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (ECC-Insight LLC, 2016). The CSM included below is a summary from the *Draft 2015 Annual Performance Monitoring Report, Enhanced In Situ bioremediation Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California*.

5.1 BACKGROUND

Background information presented here are brief description of the site conditions. Details of site conditions, previous investigations, and background information are discussed in Section 2 of the *Final Remedial Action Work Plan Implementation of Enhanced In Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (ECC-Geosyntec, 2008).

Groundwater at IR Site 70 is impacted up to 160 feet below ground surface (bgs) by chlorinated solvents, primarily trichloroethene (TCE) and associated TCE degradation compounds. The groundwater contamination has been divided into two areas: the source area and the downgradient dissolved-phase VOC plume extending in a south-southeast direction from the source area. The remedial approach for the site is enhanced in situ bioremediation (EISB) using emulsified vegetable oil (EVO) and the dechlorinating microbial culture KB-1® in a grid of injection wells, combined with installation of biobarriers located at the source area and in the targeted hydrogeological units. The hydrogeological units detailed in Section 5.2 were updated in 2012 as part of the updated CSM. The updated CSM is also presented graphically in Figures 4 through 14.

5.2 LITHOLOGY

This CSM consists of six separate hydrostratigraphic units or model layers. Working down from ground surface, these are:

- **Upper Fines Unit (from surface fill material to approximately 60 feet bgs)—**The Upper Fines Unit comprises the following five subunits:

  — Clay 1: Clayey soils are present from below the fill to approximately 20 feet bgs (i.e., “surficial fines”).

— Sand 1: Beginning at approximately 20 feet bgs, a predominantly fine-grained sand is present that ranges in thickness from 2 to 17 feet (i.e., "20-foot sand").

— Clay 2: Beginning at approximately 30 to 38 feet bgs, a predominantly clayey unit is present that ranges in thickness from approximately 3 to 10 feet (i.e., "30-foot fines").

— Sand 2: Beginning at approximately 38 to 57 feet bgs, a fine- to medium-grained sand unit is present that ranges in thickness from 13 to 20 feet (i.e., "40-foot sand").

— Clay 3: Beginning at approximately 57 to 60 feet bgs, a silty clay to clay layer is present that ranges in thickness from 2 to 3 feet (i.e., "60-foot clay").

- **First Sand Unit (approximately 60 to 105 feet bgs)—**The First Sand Unit consists of interbedded fine-grained and medium-grained sands and silty sands. A coarse sand/fine gravel layer was encountered in several borings between 80 and 95 feet bgs, and localized clays layers were observed between 80 to 90 feet bgs in several source area biobarrier injection wells. The First Sand Unit varies in thickness from approximately 30 to 50 feet.

- **Shell Horizon Unit (approximately 105 to 135 feet bgs)—**The Shell Horizon Unit consists of a sequence of interbedded clays, silts, sands, and gravels with laterally discontinuous interbeds of dense shells and shell fragments. In the source area, this unit comprises a sequence of interbedded clays, silts, sands, and gravels. The unit transitions to predominantly fine-grained sand near well RDO-6A/B between First and Second Street in the downgradient plume area. Within the CSM, the Shell Horizon Unit is subdivided into the Shell Horizon Clays Unit (interbedded) and Shell Horizon Sand Unit (fine-grained) to reflect differences in hydrogeologic characteristics.

- **Second Sand Unit (approximately 135 to 170 feet bgs)—**Although similar in overall lithology to the First Sand Unit, the Second Sand Unit appears to be slightly coarser grained in its upper section.

- **Deep Clay Unit (approximately 170 to 190 feet bgs)—**The Deep Clay Unit consists of an upper clay to silty clay horizon and a lower clayey silt, silt, sandy silt, or sandy clay layer. The upper clay to silty clay layer appears to be a continuous low-permeability unit that is encountered between 164 to 176 feet bgs. The upper clay to silty clay unit grades downward to clayey silt, silt, sandy silt, or sandy clay. This lower layer is 3 to 20 feet thick, and extends to depths of 175 to 188 feet bgs. Below the clayey silt, silt, sandy silt, or sandy clay layer is a silty sand and sand layer, up to 6 feet thick, which has been observed at a depth of 191 feet bgs.

- **Deep Sand Unit (approximately 190 feet bgs and deeper)—**The Deep Sand Unit consists of fine-grained sands and silty sands and appears to be similar in character to the First and Second Sand Units. It should be noted that the Deep Sand Unit has been logged in only a few boreholes on NAVWPNSTA Seal Beach.

5.3 EXTENT OF CONTAMINATION

The spatial distribution of TCE exceeding 250 micrograms per liter (µg/L) in groundwater at IR Site 70 was approximately 4,000 feet by 2,200 feet as reported in the *Final Remedial Design for IR Site 70 Enhanced In Situ Bioremediation, Naval Weapons Station, Seal Beach, Seal Beach, California* (Geosyntech, 2006b). This distribution is based on groundwater sampling data collected during a 2005 groundwater monitoring event and the pilot study event. The design of the EISB system was based on the vertical and lateral extent of the 250 µg/L TCE plume. Since the installation of the EISB system, TCE concentrations across the site have decreased and the TCE plume exceeding 250 µg/L is limited to be within and near the source area. The highest TCE concentrations remain in source area at the Upper Fines Unit and the First Sand Unit.

The EISB system consists of series of monitoring wells, injections wells, biobarriers, and point of compliance wells. Majority of the system wells were installed in 2007 and in 2008 groundwater from select monitoring wells were sampled for the first time to establish baseline concentrations at IR Site 70. An initial EISB injection consisting of EVO and microbial culture KB-1® were injected into source area wells and six downgradient biobarriers wells from 2008 to 2010. To date, one additional round of EVO injection (without additional microbial culture) was completed in select wells. Rationale for the locations of the additional EVO injections, completed between September and December 2013, are included in the *Final Annual Performance Monitoring Report, Enhanced In Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (Tetra Tech, 2013). Since the baseline sampling activities, groundwater at IR Site 70 has been monitored for geochemical, biogeochemical, and chemical parameters on a semiannual basis to ensure that groundwater treatment is still occurring.

As part of updating the CSM, lateral and vertical distribution of contaminants based on December 2015 monitoring event are presented in Figures 4 through 14. The concentration of each constituent of concern reported for each hydrostratigraphic unit from the December 2015 monitoring event are detailed as follows:

- Upper Fines Unit:
  - Maximum TCE concentration was detected at 2,200 µg/L (MW-70-MNA01)
  - Maximum cis-1,2-DCE concentration was detected at 5,400 µg/L (MW-70-28)
  - Maximum vinyl chloride (VC) concentration was detected at 2,400 µg/L (MW-70-28)
  - Maximum trans-1,2-DCE concentration was detected at 86 µg/L (MW-70-MNA01)

— Perchloroethylene (PCE) was not detected above the target cleanup goal (TCG) of 5 µg/L in any of the sampled wells

— Maximum 1,1-DCE was detected at 41 µg/L (MW-70-28)

— Chloroform was not detected above the TCG of 100 µg/L in any of the sampled wells

— Maximum 1,1-dichloroethane was detected at 5.5 µg/L (MW-70-28)

- First Sand Unit:

  — Maximum TCE concentration was detected at 3,400 µg/L (MW-70-PMW06B)

  — Maximum cis-1,2-DCE concentration was detected at 1,000 µg/L (MW-70-MNA12)

  — Maximum VC concentration was detected at 790 µg/L (MW-70-PMW06A)

  — Maximum trans-1,2-DCE concentration was detected at 140 µg/L (MW-70-PMW06B)

  — PCE was not detected above the TCG of 5 µg/L in any of the sampled wells

  — Maximum 1,1-DCE was detected at 8.6 µg/L (MW-70-PMW06A)

  — Chloroform was not detected above the TCG of 100 µg/L in any of the sampled wells

  — 1,1-dichloroethane was not detected above the TCG of 5 µg/L

- Shell Horizon Unit:

  — Maximum TCE concentration was detected at 7.4 µg/L (MW-70-SHB217)

  — Maximum cis-1,2-DCE concentration was detected at 1,200 µg/L (MW-70-MNA14)

  — Maximum VC concentration was detected at 290 µg/L (IW-70-SHB113)

  — Maximum trans-1,2-DCE concentration was detected at 56 µg/L (IW-70-SHB217)

  — PCE was not detected above the TCG of 5 µg/L in any of the sampled wells

  — 1,1-DCE was not detected above the TCG of 6 µg/L in any of the sampled wells

  — Chloroform was not detected above the TCG of 100 µg/L in any of the sampled wells

  — 1,1-dichloroethane was not detected above the TCG of 5 µg/L

- Second Sand Unit:
  - — Maximum TCE concentration was detected at 6.8 µg/L (MW-70-MNA16)
  - — Maximum cis-1,2-DCE concentration was detected at 1,100 µg/L (MW-70-MNA16 and MW-70-PMW14A)
  - — Maximum VC concentration was detected at 410 µg/L (MW-70-PMW14B)
  - — Maximum trans-1,2-DCE concentration was detected at 79 µg/L (MW-70-PMW14A)
  - — PCE was not detected above the TCG of 5 µg/L in any of the sampled wells
  - — 1,1-DCE was not detected above the TCG of 6 µg/L in any of the sampled wells
  - — Chloroform was not detected above the TCG of 100 µg/L in any of the sampled wells
  - — 1,1-dichloroethane was not detected above the TCG of 5 µg/L
- Deep Sand Unit:
  - — Contaminants of concern (COCs) were not detected in any of the Deep Sand wells during the 2015 monitoring event

SAP Worksheet #6: Project Quality Objectives/Systematic Planning Process Statements

6.1 STATE THE PROBLEM

An EISB system has been installed at IR Site 70 to address groundwater contamination consisting of VOCs, primarily TCE, TCE dechlorination products (1,1-dichloroethene, 1,1-dichloroethane, cis-1,2-dichloroethene, trans-1,2-dichloroethene, and VC), PCE, and chloroform. Concentrations of TCE in excess of the TCG persist in four hydrostratigraphic units: the Upper Fines Unit, the First Sand Unit, the Shell Horizon, and Second Sand Unit. TCGs are the California maximum contaminant levels (MCLs) for drinking water (Title 22 of the California Code of Regulations). The overall objective of the EISB system is to reduce COCs in groundwater at IR Site 70 to concentrations below the TCGs. The design of the EISB system was based on TCE plume of 250 µg/L. The remedial design for IR Site 70 is to terminate active EISB treatment once TCE plume in the source area has been reduced to 200 µg/L. The remainder of the TCE plume will be treated through monitor natural attenuation (MNA) until final cleanup goals are achieved. Active EISB treatment is defined when continued re-injection of EVO is required. EVO was last injected in 2013. Continued operational and performance monitoring are necessary to assess the condition of EISB system (i.e., anaerobic condition) and the progress of TCE breaking down to ethene.

In addition to the continued groundwater monitoring, as the EISB treatment progresses, hydrogen sulfide and methane gas can be produced as by product of the EISB process. Since installation of the EISB system, both methane and hydrogen sulfide have been monitored for H&S concerns for construction workers performing trenching activities, as methane and hydrogen sulfide may be confined in the vadose zone. Both hydrogen sulfide and methane were detected in soil vapor samples collected from the recent December 2015 monitoring event. Methane was reported above the lower explosive level (LEL) of 50,000 ppmV at five out of eleven locations. At three of the five locations the upper explosive level (UEL) of 150,000 ppmV was also exceeded. Hydrogen sulfide was detected in two of the eleven samples collected with concentrations between 0.83 and 3.5 ppmV. Hydrogen sulfide has a permissible exposure limit ceiling of 20 ppmV. Soil vapor samples were also analyzed for VOCs for the first time during the December 2015 monitoring event. VOCs, Freon 113, 1,1-dichloroethane, 1,1-dichloroethene, 1,2-dichloropropane, 1,4-dichlorobenzene, benzene, cis-1,2-dichloroethene, chloroform, trans-1,2-dichloroethene, tetrachloroethene, trichloroethene, and vinyl chloride, were detected in some soil vapor samples. The amount of VOCs confined in the vadose zone from VOC partitioning process has not been monitored in the past, except during the December 2015 event. In late 2015, the occupied building located at the source area was demolished; therefore concern for vapor intrusion in the source area has been mitigated. A potential for VOCs in the vadose zone migrating away from the source area may impact occupants in buildings located downgradient from the source area. Additional VOCs data of the vadose zone is needed to understand the fate and transport of VOCs in groundwater at the source area treatment grid.

6.2 IDENTIFY THE GOALS OF THE STUDY

The data obtained from the EISB system monitoring will be used to measure progress toward the following overall performance goal. The data will be obtained by posing the following decision questions:

- Does the data confirm a permanent reduction in the concentration of COCs, primarily TCE, in the source area to below the 200 µg/L concentration?
- Does the data confirm a permanent reduction in the concentrations of COCs, primarily TCE, in the dissolved-phase plume groundwater toward TCGs listed in Worksheet #10 and confirm that concentrations continue to meet this criterion post-treatment?
- Does the data indicate that TCGs have been achieved for all COCs in the source area and in the dissolved-plume for five years such that no further action status for each zone can be requested?
- Does the soil vapor data indicate that the methanogenic reaction is occurring and that generated methane confined in vadose zone is safe for construction workers performing trenching activity?
- Is hydrogen sulfide present in the vadose zone at an unsafe level for construction workers?
- Is the amount of VOCs present in the vadose zone similar each event?

6.3 IDENTIFY INFORMATION INPUTS

Groundwater samples will be collected from existing monitoring well network (Figure 2) that represents each hydrostratigraphic unit. Samples will be analyzed for the COCs and parameters used to evaluate conditions associated with EISB treatment and MNA. The analyses for groundwater include:

- Not all wells will be sampled for all parameters below (see SAP Worksheet #9)
- VOCs—EPA Method 8260B
- Dissolved gases (ethene, ethane, methane)—RSK175
- Anions (nitrate, nitrite, sulfate, chloride, bromide)—EPA Method 300.0
- Total dissolved solids (TDS)—Standard Method (SM) 2540C
- Alkalinity—SM2320B
- Volatile fatty acids (VFAs)—EPA Method 300 modified (M)
- Total organic carbon (TOC)—SM5310B

- Sulfide— SM4500S2F
- Dissolved metals (arsenic, iron, manganese)—EPA Method 6010B
- Dehalococcoides gene/VC reductase A gene (DHC/vcrA)—CENSUS
- Field parameters (pH, oxidation-reduction potential [ORP], dissolved oxygen [DO], temperature, turbidity, conductivity)

In addition, soil vapor will also be sampled to confirm whether methane, hydrogen sulfide, and VOCs are being generated as results of the EISB process. The specific wells to be sampled are listed in SAP Worksheet #9.

- Soil Vapor VOCs—Toxic Organics (TO)15
- Soil Vapor Hydrogen Sulfide—EPA 15/16
- Soil Vapor Methane Gas—ASTM D1946

6.4 DEFINE THE BOUNDARIES OF THE STUDY

Treatment and monitoring areas are defined into two lateral areas: the source area grid and the downgradient dissolved-phase VOC plume located south-southeast from the source area grid (Figure 2). Vertically, contaminated groundwater exists in four primary hydrogeological units: the Upper Fine Unit, the First Sand Unit, the Shell Horizon Unit, and Second Sand Unit. Data collected from additional hydrostratigraphic units, the Deep Clay Unit and the Deep Sand Unit, will be used to monitor further migrations. Figure 2 shows the monitoring well network for IR Site 70.

Twelve soil gas monitoring wells were installed and screened 5 to 6 feet bgs in the source area to monitor generated methane and hydrogen sulfide. Soil vapor probes were installed near injection wells, particularly near buildings that were active at the time and utility corridors. Soil vapor probe locations are shown in Figure 3.

The next semiannual groundwater sampling activity will begin in December 2016 and continue semiannually until 2018. No sensitive species inhabit the site and normal weather patterns should not affect monitoring activities.

6.5 DEVELOP THE ANALYTIC APPROACH

The following decisions will be made based on the data collected:

- **If** TCE concentrations are reduced to 200 µg/L in the source area, **then** switching remediation to MNA will be evaluated.

- **If** TCE concentrations exceed 200 µg/L in the source area, **then** continue monitor groundwater for analytical parameters listed in Section 8.1 in the source area to evaluate whether additional EVO or microbial culture injections may be needed.

- **If** TCE concentrations in the downgradient dissolved-phase are decreasing toward the TCG, and TCE concentrations in source area are less than 200 µg/L, **then** remediation will be switched to MNA.

- **If** TCE concentrations in the downgradient dissolved-phase plume are not decreasing toward the TCG, **then** groundwater monitoring will continue and data will be evaluated to determine whether additional EVO or microbial culture injections may be needed.

- **If** TCGs are not achieved by all COCs, including chloroform and all TCE breakdown products, in the desired timeframe previously identified in the *Final Remedial Design for IR Site 70 Enhanced In-Situ Bioremediation, Naval Weapons Station, Seal Beach, Seal Beach, California* (Geosyntech, 2006b), **then** potential modifications of the remedial actions will be evaluated.

- **If** all COCs concentrations, including chloroform and all TCE breakdown products, are decreasing toward meeting the TCGs, **then** MNA will continue until TCGs are achieved.

- **If** TCGs are achieved by all COCs and through interactive process taking into account site-specific factors such as aquifer classification and designated use, and MNA performance, **then** MNA will be terminated in a no further action required status for each zone.

6.5.1 Soil Vapor

- **If** methane concentrations in soil vapor samples are reported as detected, **then** data concentrations trend chart will be produced to monitor methane concentrations in the source area for conditions between the LEL and UEL, which may impact construction workers performing trenching activity.

- **If** methane concentrations in all soil vapor samples are reported as non-detected, then methane data in groundwater will be reviewed to confirm methanogenic reactions are not occurring.

- **If** hydrogen sulfide is detected in the soil vapor samples, **then** hydrogen sulfide concentrations will be monitored for unsafe conditions that may affect construction workers**.**

- **If** hydrogen sulfide is not detected in the soil vapor samples for five years, **then** request to end hydrogen sulfide analysis may be recommended.

- **If** VOCs concentrations in soil vapor samples are similar for each event, **then** data may indicate that VOCs partitioning has reached equilibrium, and the potential for VOCs in the vadose zone transporting away from the source is minimum.

- **If** VOCs concentrations in soil vapor samples are not similar for each event, **then** data would seem to indicate that VOCs partitioning has not reached equilibrium and additional soil vapor monitoring events may be recommend to determine the fate and transport of VOCs in the source area.

6.6 SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA

To limit uncertainty in the obtained environmental data, criteria for the precision, accuracy, representativeness, completeness, and comparability parameters and limit of detection (LOD) for the have been developed by the laboratory. Measurement errors will be controlled by using appropriate sampling and analytical methods. Laboratory errors will be controlled by adhering to the *Quality System Manual for Environmental Laboratories, Version 5.0* (QSM; U.S. Department of Defense [DoD], 2013), following established standard operating procedures (SOPs), and performing independent data validation to verify laboratory processes. The field crews will review the SAP before sample collection to limit sample collection errors. The subcontracted analytical laboratory will have a copy of this SAP and will adhere to DoD QSM guidance to limit measurement errors.

6.7 DEVELOP THE PLAN FOR OBTAINING DATA

The sampling strategy for both groundwater and soil vapor monitoring is described in Worksheet #8. Groundwater samples in the treatment areas will be collected from existing wells installed in the source area and the downgradient dissolved-phase VOC plume area. Specific wells to be sampled throughout the treatment period are listed in Worksheet# 9.1. Groundwater samples will be collected on semiannual basis until groundwater data indicate a change in sampling frequency is necessary or no further monitoring is needed. The groundwater sampling program will allow for continuing monitoring of TCE degradation progress by the EISB remedial activities.

In addition to groundwater monitoring, soil vapor at IR Site 70 will also be monitored for methane, hydrogen sulfide, and VOCs. All soil vapor probes will be sampled and analyzed for methane and hydrogen sulfide. However, soil vapor samples from probes located at perimeters of the source area will be analyzed for VOCs. Soil vapor will be sampled and analyzed annually from vapor probes listed in Worksheet 9.1.

SAP Worksheet #7: Measurement Performance Criteria Table—Field Quality Control Samples (Groundwater)

QC Sample	Analytical Group	Frequency	Data Quality Indicator (DQI)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicates	VOCs	One per 10 field samples collected (10 percent [%])	Precision	30% Relative percent difference (RPD)	S&A
Rinse Blanks	VOCs	1 per day for each day that non-dedicated sampling equipment is used	Representative-ness Bias Contamination	Analytes not detected above limit of quantitation (LOQ)	S
Matrix spikes (MSs) and Matrix spike duplicates (MSDs)	VOCs Dissolved Hydrogen Gases (DHGs) VFAs Anions TDS Alkalinity TOC Sulfide Dissolved Metals	One per 20 field samples collected (5%)	Precision and Accuracy	DoD QSM (2013) control limits	A
Trip Blanks	VOCs	One per sampling cooler containing VOC samples	Representative-ness Bias Contamination	Analytes not detected above LOQ	S
Temperature Blanks	VOCs DHGs VFAs Anions TDS Alkalinity TOC Sulfide Dissolved Metals	Every cooler shipped to the laboratory	Representative-ness	0 to 6 degrees Celsius (°C)	S

SAP Worksheet #8: Sampling Design and Rationale

Professional judgment was used to design the monitoring and injection well networks for the EISB treatment system and for the sampling frequency schedule present in Worksheet 9.1.1 and #9.1.2, based on previously collected data from past site investigations and groundwater monitoring events performed to date. To provide a cost-effective performance monitoring design, groundwater samples and water level elevations will be obtained using existing monitoring wells and the frequency of sampling will be reduced over time as the effective performance of the system is demonstrated. As recommended by past Annual Groundwater Monitoring Reports and based on professional judgment, analytical program was optimized based on available analytical data. Groundwater sampling and analysis optimization will continue for future monitoring events and recommendations will be included in the Annual Groundwater Monitoring Reports. Remedial activity optimization steps will follow guidelines described in the *NAVFAC Guidance for Optimizing Remedial Action Operation* (Naval Facilities Engineering Command, 2012). The sampling scheme has been designed to emphasize the measurement of TCE in groundwater samples, the primary indication of progress toward the overall performance goal. Other measurements will provide information about factors that will affect the ability of the treatment systems to achieve this goal and will enable adjustments to be made to optimize the operation of the systems.

In addition to groundwater monitoring, soil vapor will be monitored annually for VOCs, hydrogen sulfide, and methane gas. Twelve soil probes screened to 5 to 6 feet bgs were installed near the source area injection wells, particularly in the vicinity of buildings or utility corridors. Monitoring probes were installed to monitor possible hydrogen sulfide and methane gas being generated from the EISB treatment and generated gas being confined in the vadose zone that could impact construction workers. Both hydrogen sulfide and methane gas have been sampled since the installation of the EISB. Both methane and hydrogen sulfide were detected in soil gas samples collected during the recent December 2015 monitoring event. Methane was detected between 220 parts per million by volume (ppmV) and 520,000 ppmV. The LEL for methane is 50,000 ppmV and the UEL is 150,000 ppmV. Methane was reported above its LEL at five locations, including SV-70-02, SV-70-05, SV-70-06, SV-70-07, and SV-70-10. At three of the five locations the UEL was also exceeded. Hydrogen sulfide was detected in two of the eleven samples collected with concentrations between 0.83 and 3.5 ppmV. Hydrogen sulfide is regulated by the Occupational Safety and Health Administration (OSHA) and has a permissible exposure limit ceiling of 20 ppmV. Soil vapor from the December 2015 event was also analyzed for VOCs for the first time. Per the *Draft 2015 Annual Performance Monitoring Report, Enhanced In Situ bioremediation Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California* (ECC-Insight LLC, 2016), methane gas and hydrogen sulfide are monitored for H&S purposes. The VOCs data in soil vapor were collected to monitor the fate and transport of VOCs in groundwater and in vadose zone.

8.1 GROUNDWATER MONITORING PROGRAM

The groundwater sampling program measures various parameters necessary to evaluate progress toward achieving the complete reductive dechlorination of TCE. The concentrations of chlorinated ethenes, including cis-1,2-DCE and VC, serve as direct evidence of the degradation of TCE. Other performance indicators include changes in dissolved gases and polymerase chain reaction assays specific to the microbial cultures. In addition, presence of electron donor can be assess through evaluating TOC and VFAs (acetic, butyric, lactic, and propionic acids). Determining the redox state of the aquifer at each biobarrier is important since reductive dechlorination of TCE by microbial cultures occurs best when the environment is reducing. The redox state of the aquifer is best evaluated using several parameters including ORP, DO, nitrate, ferrous iron, sulfate, and methane. Reducing environments are anaerobic as heterotrophic bacteria utilize the oxygen in the aquifer. Evaluating these parameters helps determine the aquifer's redox potential. To achieve overall performance objective of the EISB system at IR Site 70, groundwater samples will be collected in June (semiannual event) and in December (annual event). Based on optimized sampling program to date, samples from wells may be analyzed for some of the following parameters, if not all, by appropriate established methods. The groundwater monitoring program is summarized in Worksheets #9.1 and #9.2 for the semiannual and annual events, respectively.

- VOCs by EPA Method 8260B
- DHGs (ethene, ethane, and methane) by RSK175
- Anions (nitrate, nitrite, bromide, chloride, and sulfate) by EPA Method 300.0
- TDS by SM 2450C
- Alkalinity by SM2320B
- VFAs (acetic, butyric, lactic, propionic) by EPA Method 300M
- TOC by SM5310B
- Dissolved hydrogen sulfide by SM4500S2F
- Dissolved metals (iron, manganese, arsenic) by EPA Method 6010B
- Dhc/VCr by Microbial Insight's CENSUS method

8.1.1 Groundwater Sampling Using Low-Flow Purging Technique

Groundwater sampling will be performed in accordance with the procedures described in this section. Groundwater monitoring wells will be sampled for specific parameters detailed in Worksheet 9.1.1 and 9.1.2. The wells will be purged and sampled, by a low-flow purging

technique, using portable or dedicated bladder pumps for each well. The following standard procedures will be followed when sampling a monitoring well:

1. Don appropriate personal protective equipment.
2. Confirm the well identification at each monitoring/observation well.
3. Calibrate field instruments in accordance with the manufacturer's directions. Record all calibration documentation in the field logbook.
4. Measure the depth to water at each well using an electronic water level indicator probe. Record the water level measurement to the nearest 0.01 of a foot in the field logbook. Decontaminate the water level indicator before each measurement.
5. Set the intake to the pump at the approximate middle of the screen interval. The initial pump speed will be set at approximately 100 ml/min until the well recovery rate can be established so that the water column in the well does not drop more than 0.3 foot below the initial water level reading. Using a low-flow purge technique, the flow rates should range between 100 and 500 milliliters (mL) per minute. Slower purge rates may be necessary to maintain drawdown requirements.
6. Monitor water quality parameters (i.e., DO, conductivity, pH, ORP, and temperature) every three to five minutes during purging. Use in-line monitoring equipment to increase the reading stability. Record the water quality parameters on the groundwater sampling log form (Appendix A). If the water quality parameters are stable for three consecutive readings, collect samples for chemical analysis. Stabilization is achieved if successive readings are within plus or minus (±) 0.1 pH units, plus or minus 1°C for temperature, 3 percent conductivity, ±10 millivolt ORP, and 10 percent DO readings. If DO readings are less than 3 milligrams per liter, then stability criteria will be ±0.3 milligram per liter. Turbidity readings will be collected but will not be used as a stabilization parameter. If the water quality parameters have not stabilized, continue purging until stabilization occurs or three calculated well volumes have been purged.
7. Do not stop the pump after stabilization and prior to sample collection.
8. Collect samples from the discharge of the pump; fill the appropriate sample containers, as shown in Worksheet 9.2. Collect field QC samples (e.g., field duplicates) as required. Volatile organic analysis vials for VOC analysis will be filled leaving no headspace (bubbles) in the vial. Dissolved metals samples will be collected by field filtering groundwater using an in-line disposable 0.45µm filter.
9. Label, package, and prepare the samples for shipment to the laboratory. Transfer the samples to cold storage after collection.

8.2 SOIL VAPOR MONITORING

Due to potential methane and hydrogen sulfide being generated from the EISB treatment activities, soil vapor probes were installed near source area EVO injection wells, particularly in

the vicinity of buildings or utility corridors. Presence of methane and hydrogen sulfide in soil vapor confirms methanogenic reactions and sulfate reductions are occurring. Soil vapors samples were collected during previous groundwater monitoring events, and methane and hydrogen sulfide were detected in some soil vapor samples. Soil vapor will continue to be monitored for methane gas and hydrogen sulfide as part of H&S measures and add information to confirm plume degradation progress.

During recent December 2015 monitoring event, soil gas samples were also analyzed for VOCs. VOCs were detected in multiple samples and data were used to confirm VOCs in groundwater are partitioning into vadose zone. Additional data is needed to determine partitioning rate so that as VOCs degrades, a fate and transport observation can be made for the vadose zone and groundwater. Soil vapor samples will be collected annually and analyzed for the following parameters by specified method.

- VOCs by EPA TO-15
- Methane gas by EPA Method 15_16
- Hydrogen Sulfide by Method D1946

8.2.1 Soil Vapor probe Sampling Procedures

Soil vapor probes will be fitted at the surface with Y-connectors to allow one line for purging the well and one line for sampling. Soil vapor samples will be collected following the *Advisory—Active Soil Gas Investigation, California Environmental Protection Agency* (California Department of Toxic Substances Control, 2015) for off-site analysis summarized as follows.

1. Using a dedicated purge Summa® canister or gas tight syringe, purge three probe volumes of air at an approximate purge rate of 100 to 200 mL per minute.

2. Perform a shut-in test at every location to check for leaks in the above ground sampling train. The shut-in test will be performed according to California Department of Toxic Substances Control procedures.

3. Perform a leak test at every soil gas sample location during sample collection as described as follows: **Leak Test:** Leakage during soil gas sampling may dilute samples with ambient air and produce results that underestimate actual site concentrations or contaminate the sample with external contaminants. Therefore, leak tests will be performed during the collection of each soil gas sample to detect the presence of leakage during sampling activities. A leak test dome (plastic bag or box) will be placed over the sampling probe at the surface. Under the dome, a cotton ball (or small rag) moistened with isopropyl alcohol (or other acceptable compound) will be used as the tracer compound. Do not let the tracer compound touch the sampling apparatus. Place it under the dome, exposed to the air space. If the tracer compound is detected in the sample, the cause shall be evaluated and the sample recollected if

deemed necessary. Alternative leak check test can be performed using helium gas for field detection.

4. Obtain passivated Summa® canisters, vacuum gauge and water filters (if necessary) from the off-site laboratory. Verify the canisters have a vacuum pressure of no less than 27 inches of mercury prior to sample collection. Record the initial vacuum on the chain-of-custody and vapor sample collection log (Appendix A).

5. Attach the Summa® canister to the well using a calibrated flow controller (supplied by air lab with canisters). Open the canister valve to achieve a flow rate of 100 to 200 mL per min to start sample collection. Stop sample collection when the Summa® canister has approximately 1 to 3 inches of mercury vacuum remaining. After sample collection, close the Summa® canister valve and disconnect the filter.

6. Measure the final canisters vacuum and record the final vacuum on the chain-of-custody and vapor sample collection log (Appendix A).

8.3 INVESTIGATION-DERIVED WASTE FOR DISPOSAL

All waste generated, including well purge water and equipment decontaminated water, will be characterized for proper disposal. CB&I will coordinate with the Navy on the management of all nonhazardous, California Hazardous, and hazardous waste to ensure that the waste soil and wastewater from this project are disposed of in accordance with all federal and state regulations, and Navy policies.

A private subcontractor will be procured to remove all investigation-derived waste from the base and dispose of it properly. Original copies of the manifest and disposal notification forms will be provided to the transporter for shipment. Copies of waste manifests and receipts for the disposal of wastes will be retained and later provided in the final summary report.

8.3.1 Wastewater Sampling Procedures

Wastewater stored in 55-gallon drums or poly tanks will be collected as follows:

1. Put on new, clean, chemical-resistant gloves.

2. Obtain an unused disposable bailer for each sampling event.

3. Tie the bailer to a nylon cord.

4. Lower the bailer into the containment vessel. Allow sufficient time for the bailer to fill with water.

5. Retrieve the bailer and fill appropriate bottles for the analyses being requested.

6. Cap the bottles and wipe any moisture from the outside of the bottles.

7. Place a sample label, complete with the information described below, on each glass jar. **Waste Samples** will use the following naming convention:

   Wastewater (WW)-consecutive number

   Example: WW-001

8. Samples will be stored in a cooler on ice until transported to the laboratory.

9. Record the sample number, date, time, and description on the Chain of Custody Form (Appendix A). Write all entries in indelible black ink. Place in a sealable bag and place inside cooler.

8.4 EQUIPMENT DECONTAMINATION

Both dedicated sampling pumps and portable bladder pumps will be used for this project. Equipment decontamination of portable pumps will be necessary to prevent the introduction of extraneous material into samples, and to prevent cross-contamination between samples. Sampling equipment will be decontaminated by washing with a non-phosphate detergent such as Liquinox™ or equivalent. Decontamination water will be collected in 55-gallon U.S. Department of Transportation-approved drums or a poly-tank.

The following procedures will be used for decontamination of non-dedicated sampling equipment:

1. If mud or soil is adhering to the sampling equipment, first rinse with potable water. This step will decrease the gross contamination and reduce the frequency at which the non-phosphate detergent and water solution need to be changed.

2. Wash with the non-phosphate detergent and water solution. This step will remove remaining contamination from the equipment. Dilute the non-phosphate detergent as directed by the manufacturer.

3. Rinse with potable water. Change the water frequently.

4. Rinse with deionized water. This step will rinse any detergent solution and potable water residues. Rinsing will be done by applying the deionized water from a clean squeeze bottle (or equivalent) while holding equipment over a bucket.

5. The decontaminated equipment will be air dried or dried with paper towels prior to re-use.

Equipment decontamination water will be sampled in accordance with the procedures described in Section 8.3.1.

8.5 DATA RECORDING AND TRANSFER

This section details the requirements for data reporting and data package formats that will be provided by the laboratory.

8.5.1 Hard Copy Deliverables

All relevant raw data and documentation, including (but not limited to) logbooks, data sheets, electronic files, and final reports, will be maintained by the laboratory for at least 10 years. The laboratory will notify CB&I 30 days before disposal of any relevant laboratory records.

The hard copy data deliverable requirements for this project will be:

- Groundwater samples—90 percent Stage 2B and 10 percent Stage 4
- Waste samples—100 percent Stage 2A

8.5.2 Electronic Deliverables

The EDD will be in EQUIS format. The analytical laboratory will follow the requirements stated in the EQUIS 4 file format. Within 30 days after the final validation of analytical results, CB&I Project Chemist will review the data and determine usability per the requirements of *Environmental Work Instruction EVR.6—Environmental Data Management and Required Electronic Delivery Standards* (NAVFAC SW, 2005). After the data validation/data review are complete. CB&I will submit analytical data to the Naval Installation Restoration Information Solutions in the Naval EDD format. All data collected for this project (except waste data) will be uploaded to Naval Installation Restoration Information Solutions.

The laboratory will certify that the EDD and the hard copy reports are identical. Both the EDD and the hard copy will present results to two or three significant figures. Field information (e.g., date and time collected, sample identification) will be entered directly into the main database from the chain-of-custody record or uploaded from electronic files generated in the field.

8.6 DATA MANAGEMENT

This section describes the laboratory data management procedures for data review, verification, reporting, and validation.

All analytical data generated by the laboratory projects will be reviewed prior to reporting to assure the validity of reported data. This internal laboratory data review process will consist of data reduction, three levels of documented review, and reporting. Review processes will be documented using appropriate checklist forms, or logbooks, that will be signed and dated by the reviewer.

8.6.1 Data Reduction

Data reduction involves the mathematical or statistical calculations used by the laboratory to convert raw data to the reported data. The laboratory will perform reduction of analytical data as specified in each of the appropriate analytical methods and laboratory SOPs. For each method, all raw data results will be recorded using method-specific forms or a standardized output from each of the various instruments.

All data calculations will be verified and initialed by personnel both generating and approving them. All raw and electronic data, notebook references, supporting documentation, and correspondence will be assembled, packaged, and stored for a minimum of 10 years for future use. All reports will be held client confidential. If the laboratory is unable to store project-related data for 10 years, then it is the responsibility of the laboratory to contact CB&I to make alternative arrangements.

8.6.2 Laboratory Data Verification and Review

The laboratory analyst who generates the analytical data will have the primary responsibility for the correctness and completeness of data. Each step of this verification and review process will involve the evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of data is essential in ensuring that data of known quality are generated consistently. All data generated and reduced will follow well-documented in-house protocols.

8.6.2.1 Laboratory Level 1 Technical (Peer) Data Review

Analysts will review the quality of their work based on an established set of guidelines, including the QC criteria established in each method, in this SAP, and as stated within the laboratory QA Manual. This review will, at a minimum, ensure that the following conditions have been met:

- Sample preparation information is correct and complete
- Analysis information is correct and complete
- Appropriate SOPs have been followed
- Calculations are verified
- There are no data transposition errors
- Analytical results are correct and complete
- QC samples are within established control limits

- Blanks and laboratory control samples (LCSs) are within appropriate QC limits
- Special sample preparation and analytical requirements have been met
- Documentation is complete, for example, any anomalies and holding times have been documented and forms have been completed

8.6.2.2 Laboratory Level 2 Technical Data Review

A supervisor or data review specialist whose function is to provide an independent review of data packages will perform this review. This review will also be conducted according to an established set of guidelines and will be structured to verify the following finding of Level 1 data review:

- All appropriate laboratory SOPs have been followed
- Calibration data are scientifically sound, appropriate to the method, and completely documented
- QC samples are within established guidelines
- Qualitative identification of contaminants is correct
- Manual integrations are justified and properly documented
- Quantitative results and calculations are correct
- Data are qualified correctly
- Documentation is complete, for example, any anomalies and holding times have been documented and appropriate forms have been completed
- Data are ready for incorporation into the final report
- The data package is complete and complies with contract requirements

The Level 2 review will be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from at least 10 percent of the samples are checked back to the sample preparation and analytical bench sheets. If no problems are found with the data package, the review will be considered complete.

If any problems are found with the data package, an additional 10 percent of the sample results will be checked back to the sample preparatory and analytical bench sheets. This cycle will then be repeated either until no errors are found in the checked data set or until all data has been checked. All errors and corrections noted will be documented.

8.6.2.3 Laboratory Level 3 Administrative Quality Assurance Data Review

The Laboratory QA Manager will review 10 percent of all data packages. This review should be similar to the review as provided in Level 2, except that it will provide a total overview of the data package to ensure its consistency and compliance with project requirements. All errors noted will be corrected and documented.

8.6.3 Data Validation and Review

The data collected for primary contaminants (VOCs) will be validated by an independent third party data validation company. All other biogeochemical analyses and MNA analyses used to monitor the progress of the remediation progress will be reviewed by a CB&I Project Chemist to ensure QC criteria have been met and to establish data usability. The laboratory will provide 90 percent data packages in Stage 2B, and 10 percent data packages in Stage 4 format as requested by the CB&I Project Chemist. With the exception of waste characterization samples, data packages will be delivered Stage 2A format.

The data validation and review will be in compliance with the following guidance: DoD QSM (2013) and the QC criteria specified in this SAP. If necessary, further validation guidance will be obtained from *National Functional Guidelines for Inorganic Superfund Data Review* (EPA, 2014a), and *National Functional Guidelines for Superfund Organic Methods Data Review* (EPA, 2014b). Data will be flagged with the following data qualifiers:

- ***J qualifier*** denotes the analyte was positively identified, but the associated numerical value is estimated.
- ***R qualifier*** denotes the data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.
- ***U qualifier*** denotes the analyte was analyzed for, but not detected, at or below the reporting limit (RL).
- ***UJ qualifier*** denotes the analyte was analyzed for, but not detected. There is uncertainty associated with the RL.

8.6.3.1 Stage 2B and Stage 4 Data Review

Data review and validation requirements will follow the guidelines established in *Environmental Work Instruction 3EN2.1—Chemical Data Validation* (NAVFAC SW, 2001). The following paragraphs briefly describe what is reviewed for each validation stage applicable to this project.

For a Stage 2B data validation effort data quality is assessed by comparing the parameters listed below to the appropriate criteria (or limits) as specified in the project SAP, DoD QSM (2013), or by EPA method-specific requirements. 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 normally present in a data package.

Stage 2B data review may include the following QC elements (depending on the analysis being reviewed):

- Sample receipt and preservation
- Sample holding times
- Laboratory method blanks (MBs)
- Surrogate recoveries
- LCS/laboratory control duplicate (LCD) recoveries
- MS/MSD recoveries
- Laboratory sample duplicates RPD
- Initial calibrations (ICAL)
- Continuing calibration verification
- Field blanks
- Field duplicate

Stage 4 validation includes all of the above and the following QC elements:

- Internal standard recoveries
- Instrument tuning and system performance (gas chromatography/mass spectrometer)
- Analyte identification (e.g., spectra and chromatograms)
- Analyte quantitation (calculation check)

SAP Worksheet #9: Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory/Organization[1] (Name, Address, Contact, and Telephone #)	Backup Laboratory (Name, Address, Contact, and Telephone #)
Groundwater	VOCs DHGs Anions TDS Alkalinity VFAs TOC Sulfide Dissolved Metals	listed in Worksheet 9.1	EPA 8260B RSK175 EPA 300.0 SM2540C SM2320B EPA 300M SM5310B SM4500-S2F EPA 6010B	10 to 15 Business Days	EMAX 1835 W. 205th Street Torrance, California 90501 Richard Beauvil 310. 618.8889 x 118	Eurofins Lancaster 2425 New Holland Pike Lancaster, Pennsylvania 17605 Laura Caulfield 717.556.7354
Groundwater	DHC/vcrA	listed in Worksheet 9.1	CENSUS	15 to 21 Business Days	Microbial Insights 10515 Research Dr. Knoxville, Tennessee 37932 Jennifer Ericson 865.573.8188	SiREM 130 Research Lane, Suite 2 Guelph Ontario N1G5G3 Ximena Druar 519.822.2265
Soil Gas	VOCs Hydrogen Sulfide Methane	listed in Worksheet 9.1	TO-15 EPA 15/16 ASTM D1946	10 to 15 Business Days	TestAmerica 880 Riverside Parkway West Sacramento, California 95605 Tracy Dutton 916.373.5600	Eurofins Air Toxics 180 Blue Ravine Road, Suite B Folsom, California 95630 Brian Whittaker 916.605.3355

Notes:

[1]All analytical laboratories performing certified analyses are State of California Environmental Laboratory Accreditation Program-certified and DoD Environmental Laboratory Accreditation Program-accredited laboratories. Laboratory certifications are provided in Appendix B.

9.1 SAMPLE DETAILS TABLE

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
Source Area Treatment Grid						
IW-70-SAT35	IW-70-SAT35-Date	GW	25–55	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump
MW-70-PMW01A	MW-70-PMW01A-Date	GW	25–35	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW01B	MW-70-PMW01B-Date	GW	25–55	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-27	MW-70-27-Date	GW	25.5–35.5	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-28	MW-70-28-Date	GW	50.3–60.3	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA01	MW-70-MNA01-Date	GW	40–50	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA02	MW-70-MNA02-Date	GW	35–45	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA03	MW-70-MNA03-Date	GW	35–45	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA04	MW-70-MNA04-Date	GW	35–45	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Point of Compliance/Upper Fines						
MW-70-02	MW-70-02-Date	GW	20–30	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-03	MW-70-03-Date	GW	20–30	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
MW-70-10	MW-70-10-Date	GW	30–40	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-17	MW-70-17-Date	GW	30–40	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-22	MW-70-22-Date	GW	20–30	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Source Area Biobarrier/First Sand						
IW-70-SAB06	IW-70-SAB06-Date	GW	65–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-SAB11	IW-70-SAB11-Date	GW	65–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA05	MW-70-MNA05-Date	GW	70–80	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW02A	MW-70-PMW02A-Date	GW	70.5–80.5	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW02B	MW-70-PMW02B-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW03A	MW-70-PMW03A-Date	GW	70.5–80.5	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW03B	MW-70-PMW03B-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
First Sand Biobarrier 1						
MW-70-38	MW-70-38-Date	GW	80–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-FSB108	IW-70-FSB108-Date	GW	60–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
IW-70-FSB118	IW-70-FSB118-Date	GW	60–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW04A	MW-70-PMW04A-Date	GW	70–80	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW04B	MW-70-PMW04B-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW05A	MW-70-PMW05A-Date	GW	70–80	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW05B	MW-70-PMW05B-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW06A	MW-70-PMW06A-Date	GW	69.5–79.5	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW06B	MW-70-PMW06B-Date	GW	91–101	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
RDO-5	RDO-5-Date	GW	65–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA08	MW-70-MNA08-Date	GW	80–90	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA06	MW-70-MNA06-Date	GW	80–90	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA11	MW-70-MNA11-Date	GW	80–90	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA07	MW-70-MNA07-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-43A	MW-70-43A-Date	GW	86–106	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
First Sand Biobarrier 2						
IW-70-FSB216	IW-70-FSB216-Date	GW	65–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-FSB229	IW-70-FSB229-Date	GW	65–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump
MW-70-PMW07A	MW-70-PMW07A-Date	GW	65–75	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW07B	MW-70-PMW07B-Date	GW	85–95	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW08A	MW-70-PMW08A-Date	GW	65–75	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW08B	MW-70-PMW08B-Date	GW	85–95	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA09	MW-70-MNA09-Date	GW	64–74	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA10	MW-70-MNA10-Date	GW	75–85	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA12	MW-70-MNA12-Date	GW	75–85	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Point of Compliance/First Sand						
MW-70-11	MW-70-11-Date	GW	80–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-16	MW-70-16-Date	GW	95–105	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-35	MW-70-35-Date	GW	90–100	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
MW-70-POC01	MW-70-POC01-Date	GW	80–90	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Shell Horizon Biobarrier 1						
IW-70-SHB113	IW-70-SHB113 – Date	GW	105–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW09	MW-70-PMW09-Date	GW	120–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW10	MW-70-PMW10-Date	GW	120–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-SHB217	IW-70-SHB217-Date	GW	105–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA13	MW-70-MNA13-Date	GW	115–125	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Shell Horizon Biobarrier 3						
IW-70-SHB310	IW-70-SHB310-Date	GW	105–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-SHB311	IW-70-SHB311-Date	GW	80–95	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump
MW-70-PMW11A	MW-70-PMW11A-Date	GW	85–95	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW11B	MW-70-PMW11B-Date	GW	115–125	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW12A	MW-70-PMW12A-Date	GW	85–95	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump
MW-70-PMW12B	MW-70-PMW12B-Date	GW	115–125	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
MW-70-MNA14	MW-70-MNA14-Date	GW	115–125	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Point of Compliance/Shell Horizon						
MW-70-23	MW-70-23-Date	GW	110–130	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Second Sand Biobarrier 1						
IW-70-SSB109	IW-70-SSB109-Date	GW	125.75–165.75	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
IW-70-SSB119	IW-70-SSB119-Date	GW	125–165	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump
MW-70-PMW13A	MW-70-PMW13A-Date	GW	125–135	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW13B	MW-70-PMW13B-Date	GW	145–155	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW14A	MW-70-PMW14A-Date	GW	128–138	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-PMW14B	MW-70-PMW14B-Date	GW	149–159	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA16	MW-70-MNA16-Date	GW	141–151	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA17	MW-70-MNA17-Date	GW	140–150	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-15	MW-70-15-Date	GW	161–171	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-14	MW-70-14-Date	GW	160–170	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Portable Pump

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
MW-70-43B	MW-70-43B-Date	GW	136–146	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-MNA18*	MW-70-MNA18*-Date	GW	TBD	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Point of Compliance/Second Sand						
MW-70-21	MW-70-21-Date	GW	150–170	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-36	MW-70-36-Date	GW	150–160	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Point of Compliance/Deep Sand						
MW-70-POC02	MW-70-POC02-Date	GW	190–200	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-POC03	MW-70-POC03-Date	GW	190–200	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
MW-70-POC04	MW-70-POC04-Date	GW	195–205	Analytical Schedule is detailed in SAP Worksheet 9.1.1 and 9.1.2	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8 Dedicated Pump
Soil Vapor Samples						
SV-70-01	SV-70-01-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-02	SV-70-02-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-03	SV-70-03-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-04	SV-70-04-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8

Sampling Location	Sample ID Number	Matrix	Pump Depth (feet)	Analytical Group	Number of Samples	Sampling SOP Reference
SV-70-05	SV-70-05-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-06	SV-70-06-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-07	SV-70-07-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-08	SV-70-08-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-09	SV-70-09-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-10	SV-70-10-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-11	SV-70-11-Date	SV	5–6	Methane, hydrogen sulfide	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8
SV-70-12	SV-70-12-Date	SV	5–6	Methane, hydrogen sulfide, VOCs	Analysis quantity is detailed in SAP Worksheet 9.1.3	SAP Worksheet #8

Notes:

** An additional monitoring well may be installed if data from an ongoing investigation being performed under another contract deemed necessary.*

GW	*groundwater*
SV	*soil vapor*
TBD	*to be determined*

9.1.1 Analytical Schedule Groundwater and Soil Vapor—December 2016/2017 (Annual)

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
Source Area Treatment Grid											
IW-70-SAT35	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW01A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW01B	1	1	1	1	1	1	1	1	1	1	1
MW-70-27	1	1	1	1	1	1	1	1	1	1	1
MW-70-28	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA01	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA02	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA03	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA04	1	1	1	–	–	–	1	–	–	–	1
Point of Compliance/Upper Fines											
MW-70-02	1	1	–	–	–	–	–	–	–	–	–
MW-70-03	1	1	–	–	–	–	–	–	–	–	–
MW-70-10	1	1	–	–	–	–	–	–	–	–	–
MW-70-17	1	1	–	–	–	–	–	–	–	–	–
MW-70-22	1	1	–	–	–	–	–	–	–	–	–
Source Area Biobarrier/First Sand											
IW-70-SAB06	1	1	1	1	1	1	1	1	1	1	1
IW-70-SAB11	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA05	1	1	1	–	–	–	1	–	–	–	1
MW-70-PMW02A	1	1	1	1	1	1	1	1	1	1	1

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
MW-70-PMW02B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW03A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW03B	1	1	1	1	1	1	1	1	1	1	1
First Sand Biobarrier 1											
MW-70-38	1	1	1	1	1	1	1	1	1	1	1
IW-70-FSB108	1	1	1	1	1	1	1	1	1	1	1
IW-70-FSB118	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW04A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW04B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW05A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW05B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW06A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW06B	1	1	1	1	1	1	1	1	1	1	1
RDO-5	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA08	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA06	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA11	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA07	1	1	1	–	–	–	1	–	–	–	1
MW-70-43A	1	1	1	–	–	–	1	–	–	–	1
First Sand Biobarrier 2											
IW-70-FSB216	1	1	1	1	1	1	1	1	1	1	1
IW-70-FSB229	1	1	1	1	1	1	1	1	1	1	1

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
MW-70-PMW07A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW07B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW08A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW08B	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA09	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA10	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA12	1	1	1	–	–	–	1	–	–	–	1
Point of Compliance/First Sand											
MW-70-11	1	1	–	–	–	–	–	–	–	–	–
MW-70-16	1	1	–	–	–	–	–	–	–	–	–
MW-70-35	1	1	–	–	–	–	–	–	–	–	–
MW-70-POC01	1	1	–	–	–	–	–	–	–	–	–
Shell Horizon Biobarrier 1											
IW-70-SHB113	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW09	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW10	1	1	1	1	1	1	1	1	1	1	1
IW-70-SHB217	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA13	1	1	1	–	–	–	1	–	–	–	1
Shell Horizon Biobarrier 3											
IW-70-SHB310	1	1	1	1	1	1	1	1	1	1	1
IW-70-SHB311	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW11A	1	1	1	1	1	1	1	1	1	1	1

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
MW-70-PMW11B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW12A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW12B	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA14	1	1	1	–	–	–	1	–	–	–	1
Point of Compliance/Shell Horizon											
MW-70-23	1	1	–	–	–	–	–	–	–	–	–
Second Sand Biobarrier 1											
IW-70-SSB109	1	1	1	1	1	1	1	1	1	1	1
IW-70-SSB119	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW13A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW13B	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW14A	1	1	1	1	1	1	1	1	1	1	1
MW-70-PMW14B	1	1	1	1	1	1	1	1	1	1	1
MW-70-MNA16	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA17	1	1	1	–	–	–	1	–	–	–	1
MW-70-15	1	1	1	–	–	–	1	–	–	–	1
MW-70-14	1	1	1	–	–	–	1	–	–	–	1
MW-70-43B	1	1	1	–	–	–	1	–	–	–	1
MW-70-MNA18*	1	1	1	1	1	1	1	1	1	1	1
Point of Compliance/Second Sand											
MW-70-21	1	1	–	–	–	–	–	–	–	–	–
MW-70-36	1	1	–	–	–	–	–	–	–	–	–

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
Point of Compliance/Deep Sand											
MW-70-POC02	1	1	–	–	–	–	–	–	–	–	–
MW-70-POC03	1	1	–	–	–	–	–	–	–	–	–
MW-70-POC04	1	1	–	–	–	–	–	–	–	–	–
Field QC Samples											
MW-70-SSB109	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-PMW07A	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-MNA01	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-PMW04A	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-PMW-02A	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-PMW09	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-10	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-PMW12B	–	Field Duplicate	–	–	–	–	–	–	–	–	–
MW-70-02	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	–
MW-70-03	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	–
MW-70-35	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	–
MW-70-POC01	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	–

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)	Sulfide (4500 S2F)	Diss. Metals (6010B)	DHC/vcrA (CENSUS)
Blanks	–	Trip Blank	–	–	–	–	–	–	–	–	–
Number of Groundwater Samples Per Event[1]		99	67	47	47	47	67	47	47	47	63

Soil Vapor Samples

Well ID	Methane (ASTM D1946)	Hydrogen Sulfide (EPA15/16)	VOCs (TO-15)
SV-70-01	1	1	1
SV-70-02	1	1	–
SV-70-03	1	1	1
SV-70-04	1	1	–
SV-70-05	1	1	1
SV-70-06	1	1	–
SV-70-07	1	1	–
SV-70-08	1	1	1
SV-70-09	1	1	1
SV-70-10	1	1	–
SV-70-11	1	1	–
SV-70-12	1	1	1
Field Duplicate Samples			
SV-70-02	1	1	–
SV-70-05	1	1	1
Total Soil Vapor Samples	14	14	7

Notes:

** An additional monitoring well may be installed if data from an ongoing investigation being performed under another contract deemed necessary.*

[1] Number of samples that will be collected per event is dependent on number of trip blanks and equipment blanks that will be collected. Trip blanks are shipped with each cooler containing volatile organic analysis vials per day. Equipment blanks will be collected for days where portable pump is used.

—	*not analyzed*
ALK	*Alkalinity*
Anions	*Nitrate, nitrite, chloride, sulfate, bromide*
dissolved metals	*iron, manganese, and arsenic*
FP	*field parameter (pH, DO, ORP, specific conductance, temperature, and turbidity)*
sulfide	*total dissolved hydrogen sulfide*

9.1.2 Analytical Schedule—July 2017/2018 (Semiannual)

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)
Source Area Treatment Grid								
IW-70-SAT35	1	–	–	–	–	–	–	–
MW-70-PMW01A	1	1	1	1	1	1	1	1
MW-70-PMW01B	1	1	1	1	1	1	1	1
MW-70-27	1	1	1	1	1	1	1	1
MW-70-28	1	1	1	1	1	1	1	1
Source Area Biobarrier/First Sand								
IW-70-SAB06	1	1	1	1	1	1	1	1
IW-70-SAB11	1	–	–	–	–	–	–	–
MW-70-PMW02A	1	–	–	–	–	–	–	–
MW-70-PMW02B	1	–	–	–	–	–	–	–
MW-70-PMW03A	1	1	1	1	1	1	1	1
MW-70-PMW03B	1	1	1	1	1	1	1	1
First Sand Biobarrier 1								
MW-70-38	1	1	1	1	1	1	1	1
IW-70-FSB108	1	1	1	1	1	1	1	1
IW-70-FSB118	1	1	1	1	1	1	1	1
MW-70-PMW04A	1	1	1	1	1	1	1	1
MW-70-PMW04B	1	1	1	1	1	1	1	1
MW-70-PMW05A	1	1	1	1	1	1	1	1
MW-70-PMW05B	1	1	1	1	1	1	1	1

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)
MW-70-PMW06A	1	1	1	1	1	1	1	1
MW-70-PMW06B	1	1	1	1	1	1	1	1
RDO-5	1	–	–	–	–	–	–	–
MW-70-MNA08	1	–	–	–	–	–	–	–
MW-70-MNA07	1	–	–	–	–	–	–	–
First Sand Biobarrier 2								
IW-70-FSB216	1	1	1	1	1	1	1	1
IW-70-FSB229	1	–	–	–	–	–	–	–
MW-70-PMW07A	1	1	1	1	1	1	1	1
MW-70-PMW07B	1	1	1	1	1	1	1	1
MW-70-PMW08A	1	1	1	1	1	1	1	1
MW-70-PMW08B	1	1	1	1	1	1	1	1
MW-70-MNA09	1	–	–	–	–	–	–	–
Shell Horizon Biobarrier 1								
IW-70-SHB113	1	1	1	1	1	1	1	1
MW-70-PMW09	1	1	1	1	1	1	1	1
MW-70-PMW10	1	1	1	1	1	1	1	1
Shell Horizon Biobarrier 3								
IW-70-SHB310	1	–	–	–	–	–	–	–
IW-70-SHB311	1	–	–	–	–	–	–	–
MW-70-PMW11A	1	1	1	1	1	1	1	1
MW-70-PMW11B	1	1	1	1	1	1	1	1

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)
MW-70-PMW12A	1	1	1	1	1	1	1	1
MW-70-PMW12B	1	1	1	1	1	1	1	1
Second Sand Biobarrier 1								
IW-70-SSB109	1	–	–	–	–	–	–	–
IW-70-SSB119	1	1	1	1	1	1	1	1
MW-70-PMW13A	1	1	1	1	1	1	1	1
MW-70-PMW13B	1	1	1	1	1	1	1	1
MW-70-PMW14A	1	1	1	1	1	1	1	1
MW-70-PMW14B	1	1	1	1	1	1	1	1
MW-70-MNA16	1	–	–	–	–	–	–	–
MW-70-MNA17	1	–	–	–	–	–	–	–
MW-70-14	1	1	1	–	–	–	–	–
Field QC Samples								
MW-70-SSB109	–	Field Duplicate	–	–	–	–	–	–
MW-70-PMW07A	–	Field Duplicate	–	–	–	–	–	–
MW-70-MNA01	–	Field Duplicate	–	–	–	–	–	–
MW-70-PMW04A	–	Field Duplicate	–	–	–	–	–	–
MW-70-PMW11A	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD
MW-70-PMW12A	–	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD	MS/MSD

Well ID	FP	VOCs (8260B)	DHGs (RSK 175)	Anions (300.0)	TDS (2540C)	Alk (2320B)	VFAs (300.0M)	TOC (5310B)
Number of Groundwater Samples Per Event[1]	48	40	36	35	35	35	35	35

Notes:

** An additional monitoring well may be installed if data from an ongoing investigation being performed under another contract deemed necessary.*

[1] Number of samples that will be collected per event is dependent on number of trip blanks and equipment blanks that will be collected. Trip blanks are shipped with each cooler containing volatile organic analysis vials per day. Equipment blanks will be collected for days where portable pump is used.

—	*not analyzed*
ALK	*Alkalinity*
Anions	*Nitrate, nitrite, chloride, sulfate, bromide*
dissolved metals	*iron, manganese, and arsenic*
FP	*field parameter (pH, DO, ORP, specific conductance, temperature, and turbidity)*
sulfide	*total dissolved hydrogen sulfide*

9.2 SAMPLE COLLECTION, HOLDING TIME, AND ANALYTICAL STANDARD OPERATING PROCEDURE REQUIREMENTS TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Sample Volume	Container (number, Size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)	Data Package Turnaround Time
Groundwater	VOCs	Preparation: EPA 5030B Analysis: EPA 8260B	40 mL	3 × 40-mL vials, Teflon™-lined septum	Hydrochloric acid to pH less than (<) 2; Cool to less than or equal to (≤) 6 ˚C; no headspace	14 days	21 days
Groundwater	DHGs	Preparation and Analysis: RSK175	40 mL	3 × 40-mL vials, Teflon™-lined septum	Hydrochloric acid to pH < 2; Cool to ≤ 6 ˚C; no headspace	14 days	21 days
Groundwater	Anions	Preparation and Analysis: EPA 300.0	250 mL	1 × 250-mL polyethylene	Cool to ≤ 6 ˚C	Nitrate: 48 hours Nitrite: 48 hours Chloride: 14 days Sulfate: 28 days Bromide: 28 days	21 days
Groundwater	TDS	Preparation and Analysis: SM 2540C	1 liter	1 × 1000-mL polyethylene	Cool to ≤ 6 ˚C	7 days	21 days
Groundwater	Alkalinity	Preparation and Analysis: 2320B	100 mL	1 × 250-mL polyethylene	Cool to ≤ 6 ˚C	14 days	21 days
Groundwater	VFAs	Preparation and Analysis: EPA 300.0	100 mL	1 × 250-mL polyethylene	Cool to ≤ 6 ˚C	28 days	21 days
Groundwater	TOC	Preparation: EPA 415 Analysis: SM5310B	150 mL	1 × 250-mL polyethylene	Sulfuric acid to pH < 2; Cool to ≤ 6 ˚C	28 days	21 days
Groundwater	Sulfide	Preparation and Analysis: SM 4500 S2F	250 mL	1 × 500-mL polyethylene	Sodium hydroxide and zinc acetate Cool to ≤ 6 ˚C	7 days	21 days

Matrix	Analytical Group	Analytical and Preparation Method	Sample Volume	Container (number, Size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)	Data Package Turnaround Time
Groundwater	Dissolved Metals	Preparation: EPA 3050B Analysis: EPA 6010B	100 mL	1 × 250-mL polyethylene	Nitric Acid to pH <2; Cool to ≤ 6 ˚C	180 days	21 days
Groundwater	DHC/vcrA	Preparation and Analysis: CENSUS	1,000 mL	1 × 1000-mL polyethylene	Cool to ≤ 6 ˚C	48 hours	21 days
Soil Vapor	VOCs	Preparation and Analysis: TO-15	10 mL	1 x 1-Liter Summa® Canister	None	30 days	21 days
Soil Vapor	Methane	Preparation and Analysis: D1946	10 mL	1 x 1-Liter Summa® Canister	None	30 days	21 days
Soil Vapor	Hydrogen Sulfide	Preparation and Analysis: EPA 15_16	100 mL	1 x 1-Liter Tedlar Bag	None	72 hours	21 days

Notes:

mL *milliliter*

SAP Worksheet #10: Reference Limits and Evaluation Tables (Groundwater)—Volatile Organic Compounds

Analyte	CAS Number	Units	Target Cleanup Goal	Target Cleanup Goal Reference [1]	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
1,1,1,2-TETRACHLOROETHANE	630-20-6	µg/L	None	None	5	5	1	0.5
1,1,1-TRICHLOROETHANE	71-55-6	µg/L	None	None	5	5	1	0.5
1,1,2,2-TETRACHLOROETHANE	79-34-5	µg/L	None	None	5	5	1	0.5
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	µg/L	None	None	5	5	2	1
1,1,2-TRICHLOROETHANE	79-00-5	µg/L	None	None	5	5	1	0.5
1,1-DICHLOROETHANE	75-34-3	µg/L	5	California MCL	5	5	1	0.5
1,1-DICHLOROETHENE	75-35-4	µg/L	6	California MCL	5	5	1	0.5
1,1-DICHLOROPROPENE	563-58-6	µg/L	None	None	5	5	1	0.5
1,2,3-TRICHLOROBENZENE	87-61-6	µg/L	None	None	5	5	1	0.5
1,2,3-TRICHLOROPROPANE	96-18-4	µg/L	None	None	5	5	2	1
1,2,4-TRICHLOROBENZENE	120-82-1	µg/L	None	None	5	5	1	0.5
1,2,4-TRIMETHYLBENZENE	95-63-6	µg/L	None	None	5	5	1	0.5
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	µg/L	None	None	5	5	2	1
1,2-DIBROMOETHANE	106-93-4	µg/L	None	None	5	5	1	0.5
1,2-DICHLOROBENZENE	95-50-1	µg/L	None	None	5	5	1	0.5
1,2-DICHLOROETHANE	107-06-2	µg/L	None	None	5	5	1	0.5
1,2-DICHLOROPROPANE	78-87-5	µg/L	None	None	5	5	1	0.5
1,3,5-TRIMETHYLBENZENE	108-67-8	µg/L	None	None	5	5	1	0.5
1,3-DICHLOROBENZENE	541-73-1	µg/L	None	None	5	5	1	0.5
1,4-DICHLOROBENZENE	106-46-7	µg/L	None	None	5	5	1	0.5
2-BUTANONE	78-93-3	µg/L	None	None	10	10	5	2.5
2-CHLOROTOLUENE	95-49-8	µg/L	None	None	5	5	1	0.5

SAP Worksheet #10: Reference Limits and Evaluation Tables (Groundwater)—Volatile Organic Compounds (continued)

Analyte	CAS Number	Units	Target Cleanup Goal	Target Cleanup Goal Reference [1]	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
2-HEXANONE	591-78-6	µg/L	None	None	10	10	5	2.5
4-CHLOROTOLUENE	106-43-4	µg/L	None	None	5	5	1	0.5
4-METHYL-2-PENTANONE	108-10-1	µg/L	None	None	10	10	5	2.5
ACETONE	67-64-1	µg/L	None	None	10	10	5	2.5
BENZENE	71-43-2	µg/L	None	None	5	5	1	0.5
BROMOBENZENE	108-86-1	µg/L	None	None	5	5	1	0.5
BROMODICHLOROMETHANE	75-27-4	µg/L	None	None	5	5	1	0.5
BROMOFORM	75-25-2	µg/L	None	None	5	5	1	0.5
BROMOMETHANE	74-83-9	µg/L	None	None	5	5	2	1.1
CARBON DISULFIDE	75-15-0	µg/L	None	None	5	5	1	0.5
CARBON TETRACHLORIDE	56-23-5	µg/L	None	None	5	5	1	0.5
CHLOROBENZENE	108-90-7	µg/L	None	None	5	5	1	0.5
CHLOROETHANE	75-00-3	µg/L	None	None	5	5	2	1
CHLOROFORM	67-66-3	µg/L	100	California MCL	5	5	1	0.5
CHLOROMETHANE	74-87-3	µg/L	None	None	5	5	2	1
CIS-1,2-DICHLOROETHENE	156-59-2	µg/L	6	California MCL	5	5	1	0.5
DIBROMOCHLOROMETHANE	124-48-1	µg/L	None	None	5	5	1	0.5
DIBROMOMETHANE	74-95-3	µg/L	None	None	5	5	1	0.5
DICHLORODIFLUOROMETHANE	75-71-8	µg/L	None	None	5	5	2	1
ETHYLBENZENE	100-41-4	µg/L	None	None	5	5	1	0.5
HEXACHLOROBUTADIENE	87-68-3	µg/L	None	None	5	5	1	0.5
ISOPROPYLBENZENE	98-82-8	µg/L	None	None	5	5	1	0.5
METHYL TERT-BUTYL ETHER	1634-04-4	µg/L	None	None	5	5	1	0.5

SAP Worksheet #10: Reference Limits and Evaluation Tables (Groundwater)—Volatile Organic Compounds (continued)

Analyte	CAS Number	Units	Target Cleanup Goal	Target Cleanup Goal Reference [1]	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
METHYLENE CHLORIDE	75-09-2	µg/L	None	None	5	5	1	0.5
m,p – XYLENE	136777-61-2	µg/L	None	None	5	10	2	1
N-BUTYLBENZENE	104-51-8	µg/L	None	None	5	5	1	0.5
o-XYLENE	95-47-6	µg/L	None	None	5	5	1	0.5
PROPYLBENZENE	103-65-1	µg/L	None	None	5	5	1	0.5
SEC-BUTYLBENZENE	135-98-8	µg/L	None	None	5	5	1	0.5
STYRENE	100-42-5	µg/L	None	None	5	5	1	0.5
TERT-BUTYLBENZENE	98-06-6	µg/L	None	None	5	5	1	0.5
TETRACHLOROETHENE	127-18-4	µg/L	5	California MCL	5	5	1	0.5
TOLUENE	108-88-3	µg/L	None	None	5	5	1	0.5
TRANS-1,2-DICHLOROETHENE	156-60-5	µg/L	10	California MCL	5	5	1	0.5
TRICHLOROETHENE	79-01-6	µg/L	5	California MCL	5	5	1	0.5
TRICHLOROFLUOROMETHANE	75-69-4	µg/L	None	None	5	5	1	0.5
VINYL CHLORIDE	75-01-4	µg/L	0.5	California MCL	0.5	0.5	0.2	0.12
XYLENES, TOTAL	1330-20-7	µg/L	None	None	5	5	1	0.5

Notes:

[1] *TCG is based on California MCL listed in Table 64444-A of California Codes of Regulations, Title 22,* §64444 last updated in September 23, 2016.

DL *detection limit*

SAP Worksheet #10.1: Reference Limits and Evaluation Tables (Groundwater)—Remediation Performance Monitoring Parameters

Analyte	CAS Number	Units	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
Dissolved Gases								
Ethane	74-84-0	µg/L	None	Not applicable	2	2	0.64	0.32
Ethene	74-85-1	µg/L	None	Not applicable	2	2	0.6	0.3
Methane	74-82-8	µg/L	None	Not applicable	2	2	0.34	0.17
Anions and TOC								
Bromide	24959-67-9	mg/L	None	Not applicable	0.5	0.5	0.25	0.13
Chloride	16887-00-6	mg/L	None	Not applicable	0.2	0.2	0.1	0.05
Nitrate	14797-55-8	mg/L	None	Not applicable	0.1	0.1	0.05	0.025
Nitrite	14797-65-0	mg/L	None	Not applicable	0.1	0.1	0.05	0.025
Sulfate	14808-79-8	mg/L	None	Not applicable	0.5	0.5	0.25	0.13
TOC	-28	mg/L	None	Not applicable	0.2	0.2	0.5	0.25
Groundwater Chemistry								
TDS	10-33-3	mg/L	None	Not applicable	20	20	20	20
Alkalinity	Not applicable	mg/L	None	Not applicable	5	5	5	5
Lactate	50-21-5	mg/L	None	Not applicable	0.1	0.1	0.05	0.025
Acetate	64-19-7	mg/L	None	Not applicable	0.1	0.1	0.05	0.025
Propionate	79-09-4	mg/L	None	Not applicable	0.1	0.1	0.05	0.025
Butyrate	107-92-6	mg/L	None	Not applicable	0.1	0.1	0.05	0.03
Sulfide	14265-45-8	mg/L	None	Not applicable	1	1	1	1
Iron	7439-89-6	mg/L	None	Not applicable	0.10	0.10	0.020	.0101
Manganese	7439-96-5	mg/L	None	Not applicable	0.005	0.005	0.003	0.0027
Arsenic	7440-38-2	mg/L	None	Not applicable	0.50	0.50	0.250	0.103

SAP Worksheet #10.1: Reference Limits and Evaluation Tables (Groundwater)—Remediation Performance Monitoring Parameters (continued)

Analyte	CAS Number	Units	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
Microorganism and Functional Genes								
DHG/vcrA	Not applicable	Cell/L	None	Not applicable	1×10^3	1×10^3	1×10^3	1×10^3

Notes:

Cell/L	*cells per liter*
mg/L	*milligram per liter*
DL	*detection limit*

SAP Worksheet #10.2: Reference Limits and Evaluation Tables—Volatile Organic Compounds (Soil Vapor)

Analyte	CAS Number	Units	Project Action Limit [1]	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
Acetone	67-64-1	µg/m³	None	Not applicable	12	12	0.95	0.42
Benzene	71-43-2	µg/m³	None	Not applicable	1.3	1.3	0.64	0.25
Benzyl chloride	100-44-7	µg/m³	None	Not applicable	4.1	4.1	2.1	0.84
Bromodichloromethane	75-27-4	µg/m³	None	Not applicable	2.0	2.0	1.3	0.44
Bromoform	75-25-2	µg/m³	None	Not applicable	12	4.1	2.1	0.72
Bromomethane	74-83-9	µg/m³	None	Not applicable	1.3	3.9	3.1	1.30
2-Butanone (MEK)	78-93-3	µg/m³	None	Not applicable	4.1	2.4	1.2	0.59
1,3-Butadiene	106-99-0	µg/m³	None	Not applicable	2.0	1.8	0.88	0.34
Carbon disulfide	75-15-0	µg/m³	None	Not applicable	4.1	2.5	1.2	0.47
Carbon tetrachloride	56-23-5	µg/m³	None	Not applicable	3.9	5.0	3.1	0.94
Chlorobenzene	108-90-7	µg/m³	None	Not applicable	2.4	1.4	0.92	0.29
Chloroethane	75-00-3	µg/m³	None	Not applicable	1.8	2.6	2.1	0.81
Chloroform	67-66-3	µg/m³	None	Not applicable	2.5	1.5	0.97	0.46
Chloromethane	74-87-3	µg/m³	None	Not applicable	5.0	1.7	0.82	0.41
Dibromochloromethane	124-48-1	µg/m³	None	Not applicable	1.4	3.4	1.7	0.67
1,2-Dibromoethane (EDB)	106-93-4	µg/m³	None	Not applicable	2.6	6.1	1.5	0.58
1,2-Dichlorobenzene	95-50-1	µg/m³	None	Not applicable	1.5	4.8	2.4	0.78
1,3-Dichlorobenzene	541-73-1	µg/m³	None	Not applicable	1.7	4.8	2.4	0.66
1,4-Dichlorobenzene	106-46-7	µg/m³	None	Not applicable	3.4	4.8	2.4	0.89
Dichlorodifluoromethane	75-71-8	µg/m³	None	Not applicable	6.1	4.0	2.0	0.72
1,1-Dichloroethane	75-34-3	µg/m³	None	Not applicable	4.8	1.2	0.81	0.29
1,2-Dichloroethane	107-06-2	µg/m³	None	Not applicable	4.8	3.2	0.81	0.36
1,1-Dichloroethene	75-35-4	µg/m³	None	Not applicable	4.8	3.2	1.6	0.51

SAP Worksheet #10.2: Reference Limits and Evaluation Tables—Volatile Organic Compounds (Soil Vapor) (continued)

Analyte	CAS Number	Units	Project Action Limit [1]	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
cis-1,2-Dichloroethene	156-59-2	µg/m³	None	Not applicable	4.0	1.6	0.79	0.35
trans-1,2-Dichloroethene	156-60-5	µg/m³	None	Not applicable	1.2	1.6	0.79	0.40
1,2-Dichloropropane	78-87-5	µg/m³	None	Not applicable	3.2	4.6	3.7	1.1
cis-1,3-Dichloropropene	10061-01-5	µg/m³	None	Not applicable	3.2	3.6	1.8	0.47
trans-1,3-Dichloropropene	10061-02-6	µg/m³	None	Not applicable	1.6	1.8	0.91	0.40
Ethylbenzene	100-41-4	µg/m³	None	Not applicable	1.6	1.7	0.87	0.27
4-Ethyltoluene	622-96-8	µg/m³	None	Not applicable	4.6	3.9	2.0	0.92
Hexachlorobutadiene	87-68-3	µg/m³	None	Not applicable	3.6	21	13	4.6
2-Hexanone	591-78-6	µg/m³	None	Not applicable	1.8	1.6	0.82	0.36
Methyl-t-Butyl Ether (MTBE)	1634-04-4	µg/m³	None	Not applicable	1.7	2.9	0.72	0.18
4-Methyl-2-pentanone (MIBK)	108-10-1	µg/m³	None	Not applicable	3.9	3.3	1.6	0.55
Methylene Chloride	75-09-2	µg/m³	None	Not applicable	21	1.4	0.69	0.25
Styrene	100-42-5	µg/m³	None	Not applicable	1.6	1.9	0.96	0.28
1,1,2,2-Tetrachloroethane	79-34-5	µg/m³	None	Not applicable	2.9	2.7	1.4	0.47
Tetrachloroethene	127-18-4	µg/m³	None	Not applicable	3.3	2.7	1.4	0.35
Toluene	108-88-3	µg/m³	None	Not applicable	1.4	1.5	0.75	0.19
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	µg/m³	None	Not applicable	1.9	3.1	3.1	1.3
1,2,4-Trichlorobenzene	120-82-1	µg/m³	None	Not applicable	2.7	15	8.9	3.2
1,1,1-Trichloroethane	71-55-6	µg/m³	None	Not applicable	2.7	1.6	1.1	0.35
1,1,2-Trichloroethane	79-00-5	µg/m³	None	Not applicable	1.5	2.2	1.1	0.36
Trichloroethene	79-01-6	µg/m³	None	Not applicable	3.0	4.3	2.2	0.56
Trichlorofluoromethane	75-69-4	µg/m³	None	Not applicable	15	4.5	2.2	1.1
1,2,4-Trimethylbenzene	95-63-6	µg/m³	None	Not applicable	1.6	3.9	2.0	0.79

SAP Worksheet #10.2: Reference Limits and Evaluation Tables—Volatile Organic Compounds (Soil Vapor) (continued)

Analyte	CAS Number	Units	Project Action Limit [1]	Project Action Limit Reference	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
1,3,5-Trimethylbenzene	108-67-8	µg/m^3	None	Not applicable	2.2	3.9	2.0	0.61
Vinyl acetate	108-05-4	µg/m^3	None	Not applicable	4.3	2.8	1.4	0.51
Vinyl chloride	75-01-4	µg/m^3	None	Not applicable	2.0	2.0	1.0	0.31
m,p-Xylene	179601-23-1	µg/m^3	None	Not applicable	3.9	3.5	0.87	0.43
o-Xylene	95-47-6	µg/m^3	None	Not applicable	3.9	1.7	0.87	0.23

Notes:

[1] Project action limit for VOCs in soil vapor matrix are not developed at this time, as receptors are not currently identified for soil vapor at the site.

µg/m^3 *microgram per cubic meter*
DL *detection limit*

SAP Worksheet #10.3: Reference Limits and Evaluation Tables—Health and Safety Parameters (Soil Vapor)

Analyte	CAS Number	Units	Health and Safety Limit[1]	Health and Safety Reference[2]	Project Quantitation Limit Goal	Laboratory-Specific		
						LOQ	LOD	DL
Methane	74-82-8	ppmv	50,000	Occupational Safety and Health Administration	1	1	0.202	0.2
Hydrogen Sulfide	7783-06-4	ppmv	20	Occupational Safety and Health Administration	0.4	0.4	0.4	0.2

Notes:

[1] Presence of methane and hydrogen sulfide is being measured for H&S purpose. Limit provided is the lower exposure limit.

DL — *detection limit*
ppmv — *parts per million by volume*

SAP Worksheet #11: Analytical Standard Operating Procedure References Table

Lab SOP Number[1]	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Laboratory[2]	Variance to QSM
EMAX-8260	Volatile Organics by GC/MS; June 02, 2016; Rev. 10	Definitive	Groundwater—VOCs	Gas chromatography/mass spectrometry	EMAX Laboratories	N
EMAX-RSK175	Dissolved Gases; April 04, 2016; Rev. 3	Definitive	Groundwater—DHGs	Gas Chromatography	EMAX Laboratories	N
EMAX-300.0	Ion Chromatography Analysis; May 17, 2016; Rev. 11	Definitive	Groundwater—anions	Ion Chromatography	EMAX Laboratories	N
EMAX-2540C	Total Dissolved Solids; May 17, 2016; Rev. 8	Definitive	Groundwater—TDS	Gravimetric	EMAX Laboratories	N
EMAX-2320B	Alkalinity; June 22, 2015; Rev. 3	Definitive	Groundwater—alkalinity	Titration, autoanalyzer	EMAX Laboratories	N
EMAX-300M	Ion Chromatography Analysis for Organic Acids; June 18, 2015; Rev. 3	Definitive	Groundwater—VFAs	Ion Chromatography	EMAX Laboratories	N
EMAX-5310B	Total Organic Carbon; August 3, 2016; Rev. 3	Definitive	Groundwater—TOC	TOC Analyzer	EMAX Laboratories	N
EMAX-4500-S2F	Sulfide (Titrimetric, Iodine); October 6, 2015; Rev. 3	Definitive	Groundwater—sulfide	Titration	EMAX Laboratories	N
EMAX-6010	Inductive Coupled Plasma Emission Spectrometric Method for Trace Metal Analysis; May 18, 2016; Rev. 7	Definitive	Groundwater—dissolved metals	Inductively Coupled Plasma	EMAX Laboratories	N

SAP Worksheet #11: Analytical Standard Operating Procedure References Table (continued)

Lab SOP Number[1]	Title, Revision Date, and Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Laboratory[2]	Variance to QSM
Microorganism and Functional Genes						
DHC/vcrA	Quantitative Polymerase Chain Reaction (qPCR); SOP Number: DNA-qPCR; January 10, 2016; Rev. 1.2;	Definitive	Groundwater—DHC/vcrA	Real-time polymerase chain reaction system	Microbial Insight	N
Soil Vapor						
WS-MSA-0015	Determination of Low-Level Volatile Organics in Ambient Indoor Whole Air Samples Using GC/MS-Scan Mode; 07/31/2015; Rev. 1.6	Definitive	Soil Vapor—VOCs	Gas chromatography/mass spectrometry	TestAmerica—Sacramento	N
WS-GCA-0020	Determination of Fixed Gases (Reformed Gases) in Air Samples using Gas Chromatography; June 09, 2016; Rev. 1.3	Definitive	Soil Vapor—methane	Gas chromatography	TestAmerica—Sacramento	N
WS-GCA-0019	Determination of Volatile Sulfur Compounds from Stationary Sources; June 28, 2016; Rev. 1.2	Definitive	Soil Vapor—hydrogen sulfide	Gas chromatography	TestAmerica—Sacramento	N

Notes:

[1]Electronic copies of laboratory SOPs are provided in the Appendix C.

2Analytical laboratory performing groundwater and soil vapor analyses are state of California Environmental Laboratory Program approved and DoD Environmental Laboratory Accreditation Program-accredited laboratories.

SAP Worksheet #12.1: Laboratory QC Samples Table—Volatile Organic Compounds

Matrix: Groundwater
Volatile Organic Compounds
EPA Method 8260/EMAX-8260

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Check of mass spectral ion intensities using bromofluorobenzene	Prior to ICAL and ICV	Must meet the EPA method requirements before samples are analyzed m/z Required Intensity (relative abundance) mass 50 -15 to 40% of m/z 95 mass 75 -30 to 60% of m/z 95 mass 95 Base peak, 100% relative abundance mass 96 -5 to 9% of m/z 95 mass 173 <2% of m/z 174 mass 174 greater than (>) 50% of m/z 95 mass 175 -5 to 9% of m/z 174 mass 176>95% but <101% of m/z 174 mass 177-5 to 9% of m/z 176	Retune instrument and verify the tune acceptability	Lab Manager/ Analyst	Representativeness	Meets all EPA Method requirements
Five-point ICAL for target analytes, lowest standard at or near the LOQ	Prior to sample analysis	The minimum average system performance check compound RF is 0.1 for chloromethane, 1,1-dichloroethane, and bromoform and 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane	Perform maintenance and Retune instrument and verify the tune acceptability, then repeat ICAL	Analyst	Representativeness	RF > 0.1 for chloromethane, 1,1-dichloroethane, and bromoform; RF> 0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane

SAP Worksheet #12.1: Laboratory QC Samples Table—Volatile Organic Compounds (continued)

Matrix: Groundwater
Volatile Organic Compounds
EPA Method 8260/EMAX-8260

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	After ICAL	All analytes within ±20% of expected value except for the following compounds due to erratic chromatographic behavior: bormomethane, chloroethane, chloromethane, dichlorodifluoromethane but must be within ±35% of expected value.	Prepare fresh standard and rerun ICV. If that fails, optimize instrument and repeat ICAL.	Lab Manager/ Analyst	Representativeness	Less than 20% difference
Evaluation of relative retention times	Each sample	Within ±0.06 relative retention time units	Perform maintenance and reanalyze all samples analyzed since the last retention time check	Lab Manager/ Analyst	Representativeness	Within ±0.06 relative retention time units
Daily CCV	Before sample analysis and every 10 samples and at the end of the analysis sequence	Less than 20% difference for all target analytes.	Perform instrument maintenance; then repeat initial or daily calibration; Reanalyze samples with noncompliant bracketing CCVs.	Lab Manager/ Analyst	Representativeness	Less than 20% difference

SAP Worksheet #12.1: Laboratory QC Samples Table—Volatile Organic Compounds (continued)

Matrix: Groundwater
Volatile Organic Compounds
EPA Method 8260/EMAX-8260

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards	All samples	Within ± 10 seconds from retention time and Areas within .50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Lab Manager/ Analyst	Representativeness	±10 seconds from retention time and Areas within .50% to +100% of ICAL midpoint standard
MB	One per analytical batch (20 samples or less)	No target analytes greater than or equal to (≥) ½ LOQ. For common laboratory contaminants, no analytes detected greater than LOQ	Determine the source of blank contamination, If necessary, re-extract and reanalyze MB all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness Bias Contamination	No target analytes ≥½ quantitation limit (QL)
LCS/LCD	One LCS or LCS/LCD per analytical/preparation batch (20 samples or less)	DoD QSM control Limits	Identify the problem, then re-extract and reanalyze the LCS (LCS/LCD) and all associated batch samples	Lab Manager/ Analyst	Precision and Accuracy	DoD QSM control Limits

SAP Worksheet #12.1: Laboratory QC Samples Table—Volatile Organic Compounds (continued)

Matrix: Groundwater
Volatile Organic Compounds
EPA Method 8260/EMAX-8260

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD for all analytes	One MS/MSD per analytical/preparation batch (20 samples or less)	DoD QSM control Limits if available if no DoD control limits specified then use laboratory control limits	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision and Accuracy	DoD QSM control Limits if available if no DoD control limits specified then use laboratory control limits
Surrogate standards	Every sample, spiked sample, standard, and MB	1,2-Dichloroethane-d4: 70-120 4-Bromofluorobenzene: 75-120 Toluene-d8: 85-120 Dibromofluoromethane: 85-115	Identify problem; if not related to matrix interference, reanalyze the extract or re-extract and reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	1,2-Dichloroethane-d4: 70-120 4-Bromofluorobenzene: 75-120 Toluene-d8: 85-120 Dibromofluoromethane: 85-115
Method detection limit study	Once per 12.month period	Detection limits established will be below the LOQ in accordance with DoD QSM requirements	Identify the problem, then repeat the method detection limit study	Lab Manager/ Analyst	Sensitivity	Meets project RL requirements

SAP Worksheet #12.2: Laboratory QC Samples Table—Anions and Volatile Fatty Acids

Matrix: Groundwater
Anions and Volatile Fatty Acids
EPA 300.0/EMAX-300.0

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Minimum three-point ICAL	Every 6 months or when CCV fails to meet the acceptance criteria	linear—least squares regression r >0.995	Locate the source of the problem and check standard, then repeat ICAL	Lab Manager/ Analyst	Representativeness	linear—least squares regression r >0.995
ICV	Every after ICAL and every 3 months	All analytes within ±10% of expected value	Prepare fresh standard and reanalyze ICV to rule out standard degradation or inaccurate injection, then repeat ICAL	Lab Manager/ Analyst	Representativeness	All analytes within ±10% of expected value
CCV	At the beginning of analytical batch and every 10 samples thereafter and at the end of the sequence	All analytes within ±10% of expected value	Reanalyze CCV. If reanalysis is still outside acceptance limits, discontinue the analysis. Locate source of problem and repeat ICAL.	Lab Manager/ Analyst	Representativeness	All analytes within ±10% of expected value
MB	One per preparation batch (20 samples or less)	No analytes detected ≥LOD	Re-prep and reanalyze MB and all samples processed with the nonconforming MB.	Lab Manager/ Analyst	Accuracy/Bias Contamination	No analytes detected ≥LOD

SAP Worksheet #12.2: Laboratory QC Samples Table—Anions and Volatile Fatty Acids (continued)

Matrix: Groundwater
Anions and Volatile Fatty Acids
EPA 300.0/EMAX-300.0

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS or LCS/LCD pair if there is not enough sample for MS/MSD	One in the absence of MS/MSD per preparation batch (20 samples or less)	DoD QSM (2013) control limits: RPD≤ 15%	Re-prep and reanalyze LCS and all samples processed with the nonconforming LCS.	Lab Manager/ Analyst	Accuracy/Bias	Laboratory control limits RPD≤ 15%
MS/MSD	Project designated sample matrix QC. One per preparation batch (20 samples or less)	DoD QSM (2013) control limits: RPD≤ 15%	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and extract/reanalyze the sample.	Lab Manager/ Analyst	Interferences Accuracy/Bias	Laboratory control limits RPD≤ 15%

SAP Worksheet #12.3: Laboratory QC Samples Table—Dissolved Gases

Matrix: Groundwater
Dissolved Gases
RSK175/EMAX-RSK175

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Five-point initial calibration for all analytes	Initially; as needed	RSD for each analyte ≤ 20%	Perform instrument maintenance, then repeat ICAL.	Lab Manager/ Analyst	Representativeness	RSD for each analyte ≤ 20%
Second-source ICV	After initial calibration	All analytes within ±25% of expected value	Perform instrument maintenance, then repeat ICAL.	Lab Manager/ Analyst	Representativeness	All analytes within ±25% of expected value
Daily CCV	Every 12 hours and at the end of the analytical run	All analytes within ±15% of expected value	Perform instrument maintenance; then repeat ICV and reanalyze all samples since last successful calibration verification	Lab Manager/ Analyst	Representativeness	All analytes within ±15% of expected value
MB	One per analytical batch (20 samples or less)	No target analytes ≥ ½ LOQ	Determine the source of contamination, reanalyze blank, then reanalyze MB and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	No target analytes ≥ ½ LOQ
MS/MSD	MS/MSD pair only analyzed if specifically requested by client—not included in batch QC	RPD≤ 20%	Identify problem; if not related to matrix interference, evaluate LCS results; Narrate	Lab Manager/ Analyst	Precisions and Accuracy	Laboratory control limits RPD≤ 20%
LCS/LCD pair	One LCS or LCS/LCD pair per analytical/preparation batch (20 samples or less)	RPD≤ 20%	Identify the problem; reanalyze the LCS or re-extract and reanalyze the LCS and all associated batch samples	Lab Manager/ Analyst	Precisions and Accuracy	Laboratory control limits RPD≤ 20%

SAP Worksheet #12.4: Laboratory QC Samples Table—Total Organic Carbon

Matrix: Groundwater
Total Organic Carbon
SM 5310B/EMAX-5310B

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Five-point initial calibration	Prior to sample analysis	Correlation Coefficient (r^2) $\geq$0.995	Locate the source of the problem. If outliers exist, prepare fresh calibration standards and repeat ICAL.	Lab Manager/ Analyst	Representativeness	Correlation Coefficient (r^2) $\geq$0.995
ICV	After every ICAL	All analytes within $\pm$10% of expected value	Re-analyze ICV to rule out bad injection and /or standard degradation. Else, repeat calibration.	Lab Manager/ Analyst	Representativeness	Percent recovery $\pm$10% of expected value
CCV	Every 15 samples and at the end of analysis sequence	All analytes within $\pm$10% of expected value	Repeat calibration and reanalyze all samples since last successful calibration.	Lab Manager/ Analyst	Representativeness	Percent recovery $\pm$10% of expected value
MB	One per analytical batch Batch = 20 samples or less	No detects ≥ ½ LOQ	If sufficient sample volume is available, reanalyze affected samples. If volume is not sufficient, narrate	Lab Manager/ Analyst	Accuracy	No detects ≥ ½ LOQ
LCS/LCSD	One pair per analytical batch Batch = 20 samples or less	Laboratory control limits	If sufficient sample volume is available, reanalyze affected samples. If volume is not sufficient, narrate	Lab Manager/ Analyst	Accuracy/ Precision	Laboratory control limits Recovery—80%–120% RPD—≤20
MS/MSD or Sample Duplicate	One per batch if sufficient volume	Laboratory control limits	None—refer to LCS/LCSD—Flag/discuss in narrative	Lab Manager/ Analyst	Accuracy/ Precision	Laboratory control limits Recovery—70%–130% RPD—≤20

SAP Worksheet #12.5: Laboratory QC Samples Table—Sulfide

Matrix: Groundwater
Sulfide
SM 4500S^{2-}/EMAX-4500-S2F

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
MB	One per analytical batch Batch = 20 samples or less	No detects ≥ ½ LOQ	If sufficient sample volume is available, reanalyze affected samples. If volume is not sufficient, narrate	Lab Manager/ Analyst	Accuracy	No detects ≥ ½ LOQ
LCS/LCSD	One pair per analytical batch Batch = 20 samples or less	Laboratory control limits	If sufficient sample volume is available, reanalyze affected samples. If volume is not sufficient, narrate	Lab Manager/ Analyst	Accuracy/ Precision	Laboratory control limits Recovery—80%–120% RPD—≤20
MS/MSD or Sample Duplicate	One per batch if sufficient volume	Laboratory control limits	None—refer to LCS/LCSD—Flag/discuss in narrative	Lab Manager/ Analyst	Accuracy/ Precision	Laboratory control limits Recovery—80%–120% RPD—≤25

SAP Worksheet #12.6: Laboratory QC Samples Table—Dissolved Metals

Matrix: Groundwater
Dissolved Metals
EPA Method 6010/EMAX-6010

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial calibration – minimum 1 point standard and a blank	Daily initial calibration prior to sample analysis	Acceptable ICV	Correct the problem and repeat initial calibration	Lab Manager/ Analyst	Representativeness	Acceptable ICV
ICV	Daily after initial calibration	All analytes within ±10% of expected value. RSD of replicate integration: <5%	Correct the problem and repeat initial calibration	Lab Manager/ Analyst	Representativeness	Within ±10% of expected value. RSD of replicate integration: <5%
CCV	Daily before sample analysis, every 10 samples and at the end of the analysis sequence	All analytes within ±10% of expected value. RSD of replicate integration: <5%	Repeat calibration and reanalyze all samples since last successful calibration	Lab Manager/ Analyst	Representativeness	Within ±10% of expected value. RSD of replicate integration: <5%
Calibration blank	After ICAL, before daily continuing calibration verification, after every 10 samples, and at the end of the sequence	No target analytes ≥ LOD	Identify the source of blank contamination; re-prepare and reanalyze the blank,	Lab Manager/ Analyst	Representativeness	No target analytes ½ LOQ
IDL study	Initial instrument setup and after significant change	IDLs will be below the LOD	Perform maintenance, then repeat the IDL study	Lab Manager/ Analyst	Sensitivity	IDLs will be below the ½ LOQ

Matrix: Groundwater						
Dissolved Metals						
EPA Method 6010/EMAX-6010						
QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
MB	One per digestion batch Batch = 20 samples or less	No target analytes ≥ ½ LOQ or >1/10 in any sample or 1/10 regulatory limit, whichever is greater	Identify the source of blank contamination, then re-prepare and reanalyze the MB and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	No target analytes ≥ RL
Interference Check Solution	At the beginning of an analytical run	Within ±20% of expected value for spiked analytes, absolute value for nonspiked analytes < LOD	Terminate analysis, perform maintenance to correct the problem if necessary, recalibrate if necessary then reanalyze interference check solution and all affected samples	Lab Manager/ Analyst	Accuracy	Within ±20% of expected value
MS/MSD For All Analytes	One MS/MSD pair per preparation batch Batch = 20 samples or less	RPD≤ 20%	Evaluate LCS recovery; Identify problem; if not related to matrix interference then re-prepare and reanalyze the MS/MSD pair and all samples in the associated batch	Lab Manager/ Analyst	Precision and Accuracy	RPD≤ 20%

SAP Worksheet #12.6: Laboratory QC Samples Table—Dissolved Metals (continued)

Matrix: Groundwater
Metals
EPA Method 6010/EMAX-6010

QC Check	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Actions	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS or LCS/LCSD (pair if there is not enough sample for MS/MSD)	One LCS or LCS/LCSD pair per each preparation batch Batch = 20 samples or less	DoD QSM (2013) control limits: RPD≤ 20%	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples	Lab Manager/ Analyst	Precision and Accuracy	DoD QSM (2013) control limits: RPD≤ 20%
Dilution Test	One dilution test per preparation batch Batch = 20 samples or less	1:5 dilution must agree within ±10% of the original determination	Perform post-digestion spike Evaluate for matrix interference	Lab Manager/ Analyst	Accuracy	10% Difference
Post-digestion Spike Addition	When MS fails or dilution test fails	Recovery within 80% to 120% of expected results	Narrate matrix interference	Lab Manager/ Analyst	Accuracy	Recovery within 80% to 120% of expected results

Notes:

>	*greater than*
IDL	*instrument detection limit*

SAP Worksheet #13: Data Verification and Validation (Steps I and IIa/IIb) Process Table

Data Review Input	Description	Responsible for Verification	Step I/IIa/IIb[1]	Internal/External
Chain-of-custody	Chain of Custody Forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. A copy of the Chain of Custody Form will be retained in the project file, and the original and remaining copies taped inside the cooler for shipment.	Field sampling team leader or Project Chemist	Step I	Internal
Sample Receipt	The sample cooler will be checked for compliance with preservative, temperature, and packaging requirements. Sample containers will be reviewed against the chain-of-custody for agreement. Sample receipt will be documented by the laboratory on a login sheet and sample information will be entered into the Laboratory Information Management System.	Laboratory sample receiving and PM	Step I	External
Field Notes/Logbook	Field notes will be reviewed internally and placed in the project file upon project completion.	CB&I Project Chemist and Field QC Manager	Step I	Internal
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the project file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file. At the beginning of each week, and at the completion of the site work, project file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the PM will be notified to ensure action is taken.	CB&I Project Technical Manager or CB&I PM	Step I	Internal
Laboratory Data Packages	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified by the CB&I Chemist or a third party reviewer according to the data validation procedures specified in this SAP.	Laboratory PM, CB&I Project Chemist or data validation company	Step I	Internal/External
EDD	All EDDs will be verified internally by the subcontract laboratory for completeness and technical accuracy prior to submittal to CB&I. All received EDDs will be verified by CB&I and/or the validation company against the hardcopy laboratory reports.	Laboratory, CB&I Chemist and a third party data validation company	Step I	Internal/External

SAP Worksheet #13: Data Verification and Validation (Steps I and IIa/IIb) Process Table (continued)

Data Review Input	Description	Responsible for Verification	Step I/IIa/IIb[1]	Internal/External
Sampling Methods and Procedures	Ensure that the required sampling methods were used to collect project samples, any field changes or deviations are noted in the field logbook. Review field sample collection logbooks for compliance with the approved SAP.	Project Chemist and Field QC Manager	Step IIa	Internal
Holding Times	Ensure the samples were analyzed within the EPA holding times. If holding times were not met, verify that deviations were documented and proper notifications were made.	Laboratory PM	Step IIa	External
Analytes and Project QLs Met	Ensure that the required list of analytes and that project-specific QLs specified in this SAP are met and reported per project requirements.	Laboratory PM and Project Chemist	Step IIa	Internal/External
Hardcopy Data Packages	Review data package for compliance with EPA method requirements (1996, 2008) and DoD QSM (2013) requirements, and approved SAP.	Third Party Data Validation Company	Step IIa	External
Documentation of All EPA Method QC Sample Results; 90 Percent Level III Data Review	Determine if all EPA Method required QC samples were analyzed and met required control limits per SAP and DoD QSM requirements when applicable.	Third Party Data Validation Company	Step IIa	External
10 Percent Level IV Raw Data Review	Recalculation of laboratory raw data to confirm laboratory calculation of final results.	Third Party Data Validation Company	Step IIa	External
Documentation of all SAP QC Sample Results	Determine if all SAP required QC samples were collected and met required control limits per SAP and DoD QSM requirements when applicable.	Project Chemist and Third Party Data Validation Company	Step IIa	Internal/External
Sampling Plan	Determine whether the SAP was executed as specified (number, location, type of field samples collected).	Project QC Manager or Project Chemist	Step IIba	Internal
Field Duplicate Precision	Compare results for groundwater and soil gas field duplicates with criteria established in this SAP.	Project Chemist and Third Party Data Validation Company	Step IIb	Internal/External
VOCs—groundwater and soil vapor	In accordance with validation company SOPs, DoD QSM, and Stage 2B and 3 guidance detailed in the National Functional Guidelines for Superfund Organic Methods Data Review (EPA 2014a).	Project Chemist and Third Party Data Validation Company	Step IIb	Internal/External

SAP Worksheet #13: Data Verification and Validation (Steps I and IIa/IIb) Process Table (continued)

Data Review Input	Description	Responsible for Verification	Step I/IIa/IIb[1]	Internal/External
Anions, TOC, TDS sulfide, alkalinity, dissolved gases, dissolved metals	In accordance with DoD QSM (2013), and Stage 2A guidance detailed in the National Functional Guidelines for Inorganic Superfund Data Review (EPA 2014b) .	Project Chemist and Third Party Data Validation Company	Step IIb	Internal/External

Notes:

[1] IIa=compliance with methods, procedures, and contracts (Table 10, page 117, Uniform Federal Policy for Quality Assurance Plans [UFP/QAPP] [EPA, 2005])

IIb=comparison with measurement performance criteria in the SAP (Table 11, page 118, Uniform Federal Policy for Quality Assurance Plans [UFP/QAPP] [EPA, 2005])

References

California Code of Regulations, Title 22, Division 4, Chapter 15, Article 5.5, Section 64444. Updated on September 23, 2016.

California Department of Toxic Substances Control, 2015, *Advisory—Active Soil Gas Investigation, California Environmental Protection Agency*, July.

ECC-Insight LLC, 2016, *Draft 2015 Annual Performance Monitoring Report, Enhanced In Situ bioremediation Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California*, June.

ECC-Geosyntec, 2008, *Final Remedial Action Work Plan, Implementation of Enhanced In-Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California*, March.

Geosyntec, 2006a, Record of Decision / Remedial Action Plan (ROD/RAP), Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California, August.

Geosyntec, 2006b, *Final Remedial Design for IR Site 70 Enhanced In-Situ Bioremediation, Naval Weapons Station, Seal Beach, Seal Beach, California*, December.

Naval Facilities Engineering Command Southwest (NAVFAC SW), 2001, *Environmental Work Instruction 3EN2.1—Chemical Data Validation*.

NAVFAC SW, 2005, *Environmental Work Instruction EVR.6—Environmental Data Management and Required Electronic Delivery Standards*.

NAVFAC SW, 2011, *Environmental Work Instruction EVR.2—Review, Approval, Revision, and Amendment of Sampling and Analysis Plans (SAPs).*

Naval Facilities Engineering Command, 2012, *Guidance For Optimizing Remedial Action Operation*, November.

U.S. Department of Defense, 2013, *Quality System Manual for Environmental Laboratories, Version 5.0*, July.

U.S. Environmental Protection Agency (EPA), 2005, *Uniform Federal Policy for Quality Assurance Plans (UFP/QAPP).*

EPA, 2014a, *National Functional Guidelines for Inorganic Superfund Data Review*, EPA 540-R-013-001, OSWER 9355.0-131, August.

EPA, 2014b, *National Functional Guidelines for Superfund Organic Methods Data Review*, EPA 540-R-014-002, OSWER 9355.0-132, August.

Tetra Tech, 2013, *Final Annual Performance Monitoring Report, Enhanced In Situ Bioremediation, Installation Restoration Program Site 70, Naval Weapons Station Seal Beach, Seal Beach, California*, October.

FIGURES

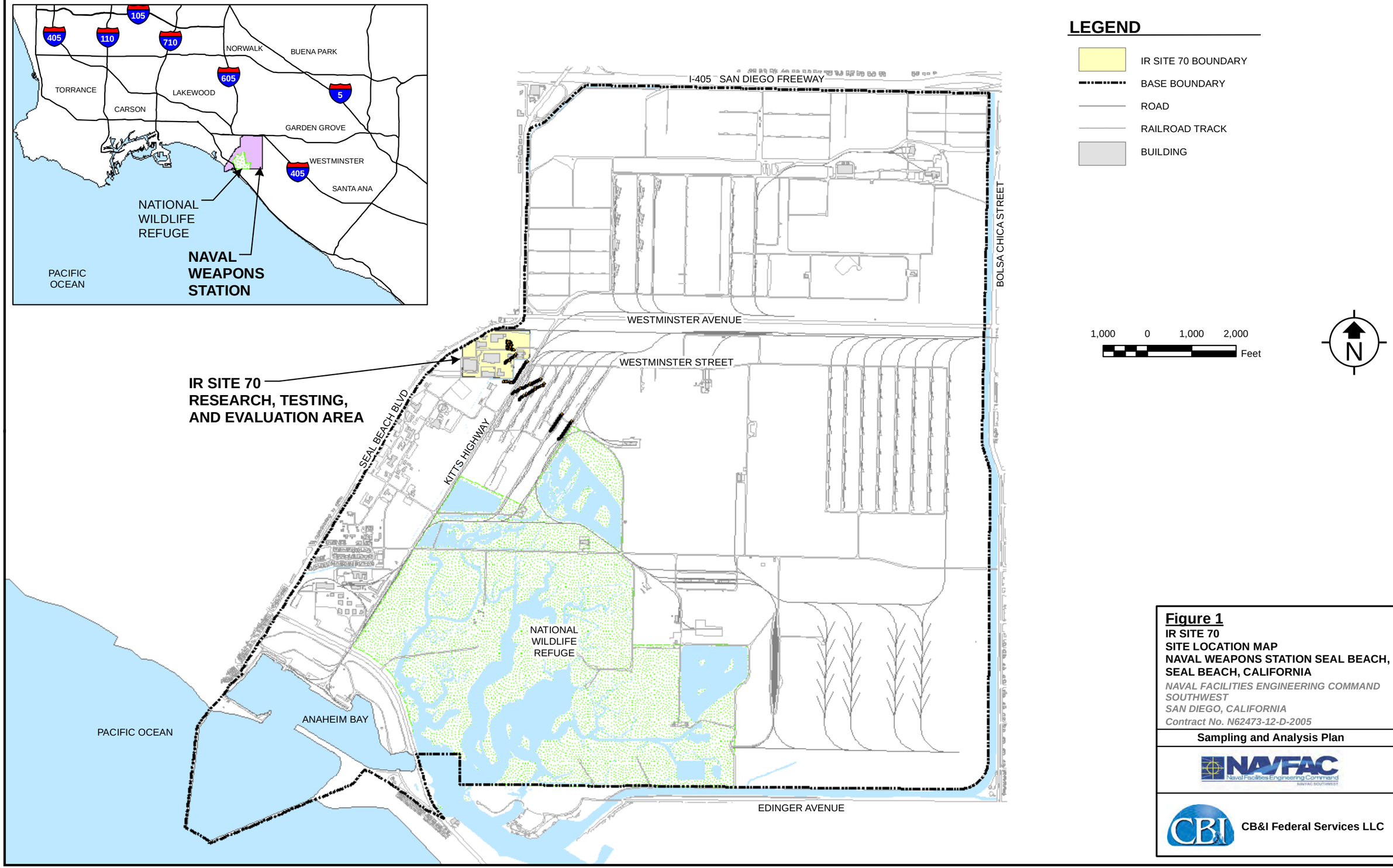


Figure 1
IR SITE 70
SITE LOCATION MAP
NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

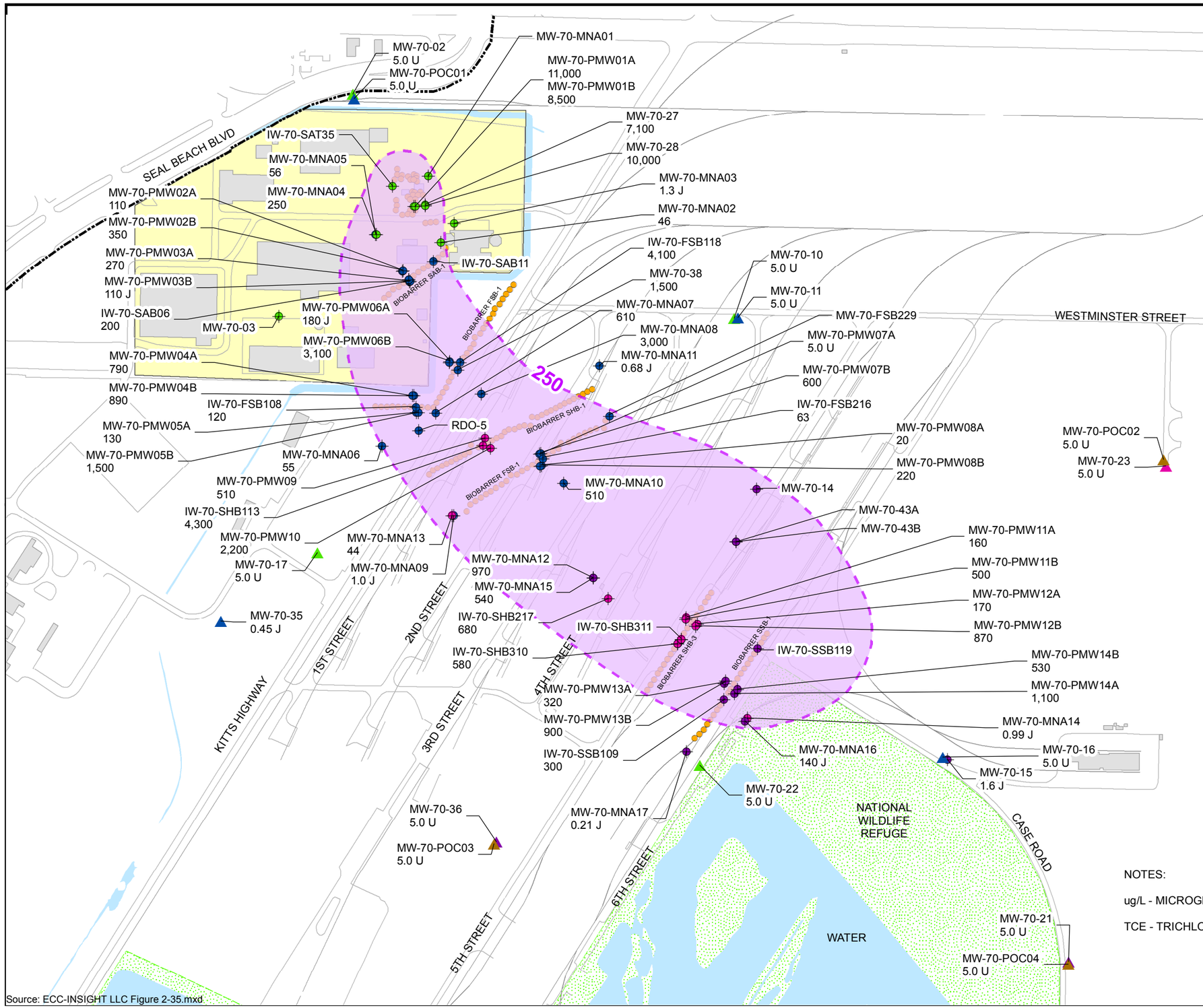
Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I **CB&I Federal Services LLC**

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 1.mxd

Source: ECC-INSIGHT LLC Figure 1-1.mxd



Source: ECC-INSIGHT LLC Figure 2-35.mxd

LEGEND

MW-70-MNA02	UPPER FINES/SOURCE AREA TREATMENT MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-22	UPPER FINES/SOURCE AREA TREATMENT POC MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-MNA08	FIRST SAND MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-11	FIRST SAND POC MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-MNA14	SHELL HORIZON MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-23	SHELL HORIZON POC MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-15	SECOND SAND MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-36	SECOND SAND POC MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
MW-70-POC02	DEEP SAND POC MONITORING WELL SHOWING WELL ID AND TCE CONCENTRATION IN ug/L
	BIOBARRIER OR SOURCE AREA TREATMENT WELL
	IR SITE 70 BOUNDARY
	BASE BOUNDARY
	ROAD
	RAILROAD TRACK
	BUILDING
250	DESIGN BASIS PLUME - APPROXIMATE EXTENT OF 250 ug/L TCE CONCENTRATION IN GROUNDWATER FALL 2008

200 100 0 200 400 Feet

N

NOTES:

ug/L - MICROGRAMS PER LITER

TCE - TRICHLOROETHYLENE

Figure 2

IR SITE 70
MONITORING WELL LOCATION NETWORK
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CBI **CB&I Federal Services LLC**

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 2_R2_TEMP_10-24-16.mxd

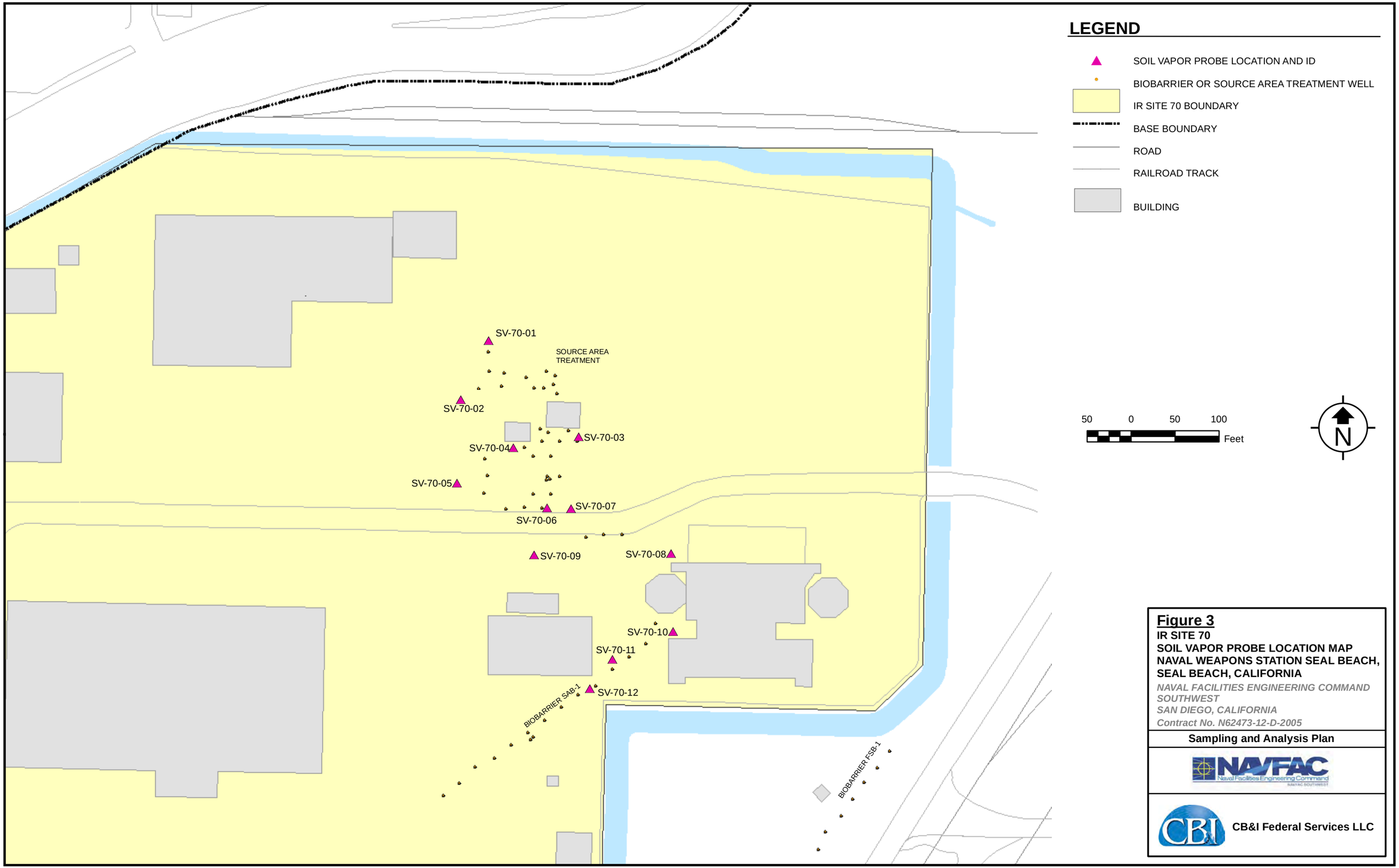


Figure 3
IR SITE 70
SOIL VAPOR PROBE LOCATION MAP
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I **CB&I Federal Services LLC**

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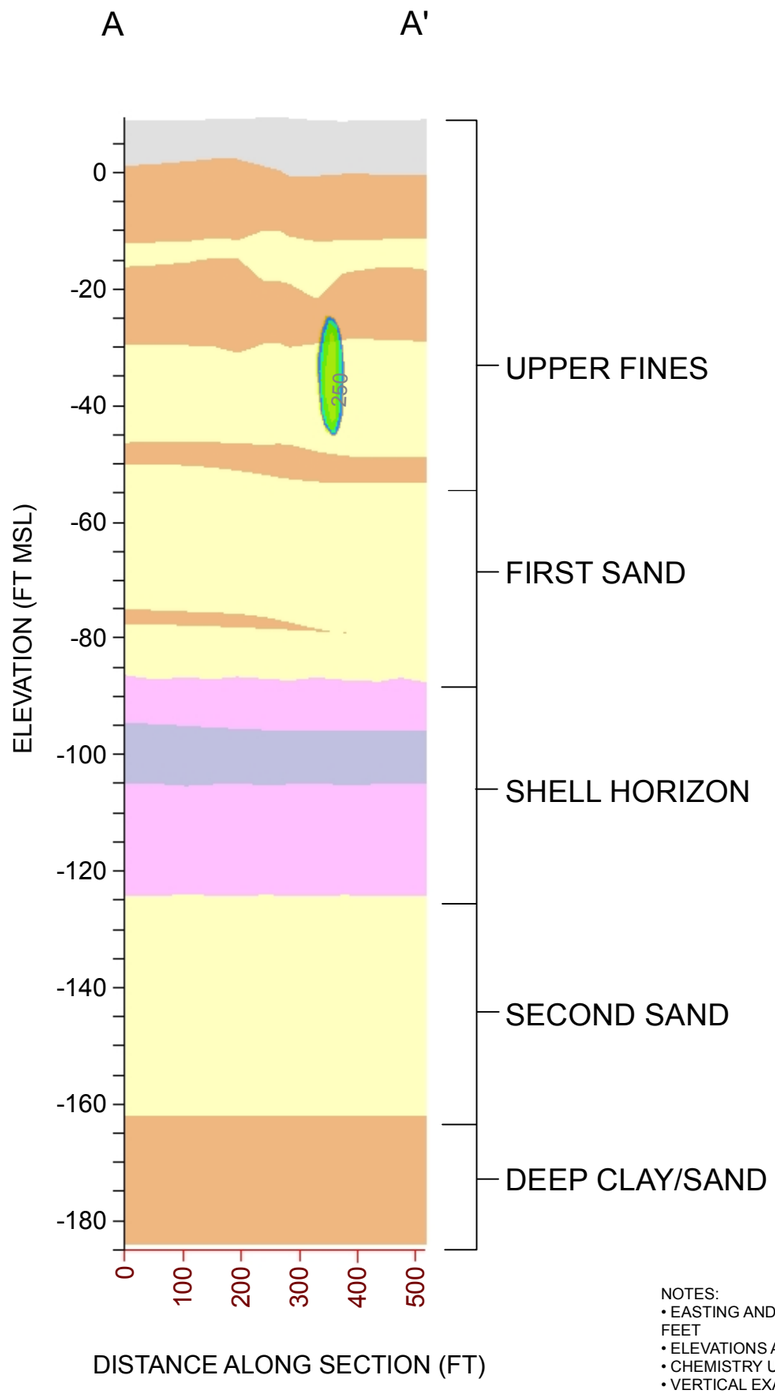
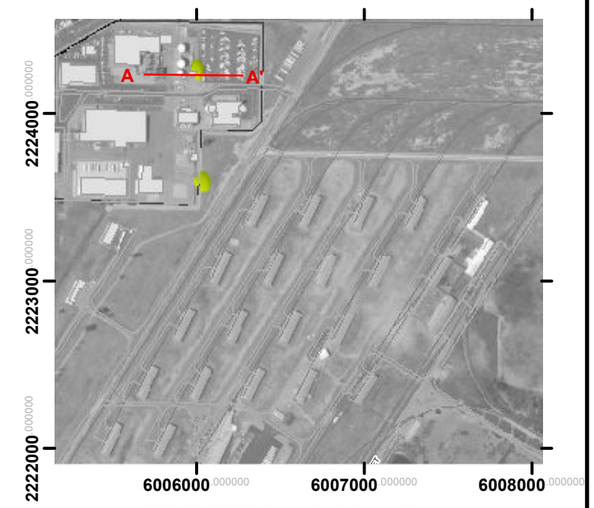
Source: ECC-INSIGHTLLC Figure 1-3.mxd



Source: ECC-INSIGHT LLC Figure 3-2.mxd

NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X



CROSS-SECTION LOCATION MAP

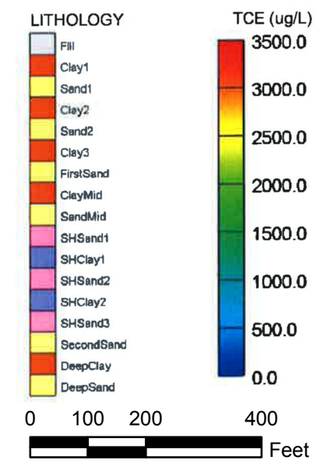


Figure 4

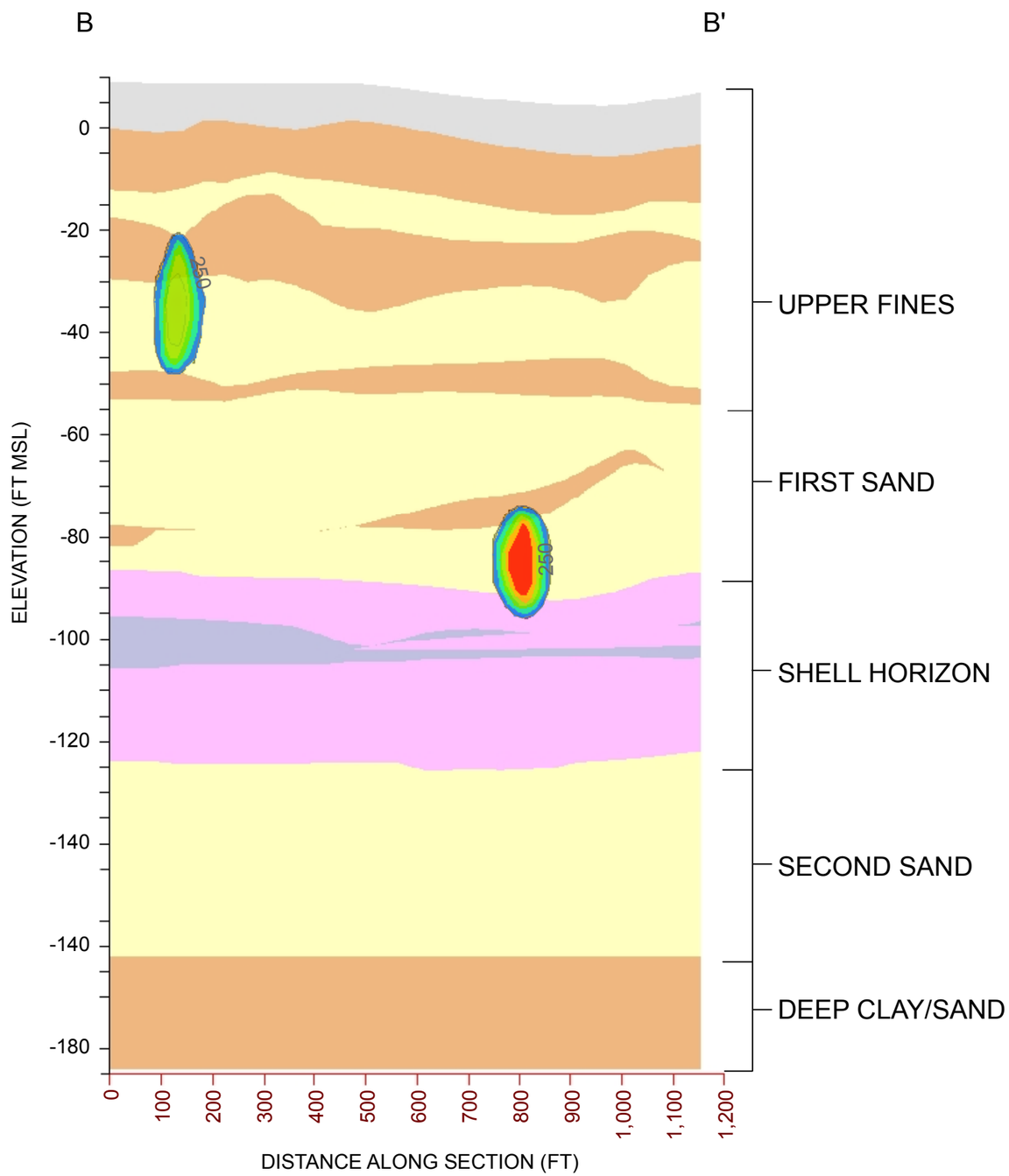
TRICHLOROETHENE EXTENT ABOVE 250 ug/L ALONG CROSSSECTION A-A',
2015 ANNUAL SAMPLING EVENT
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

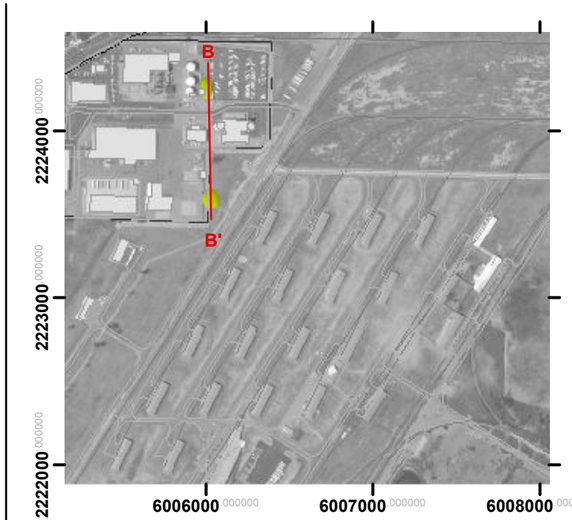
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 4 LW.mxd





CROSS-SECTION LOCATION MAP

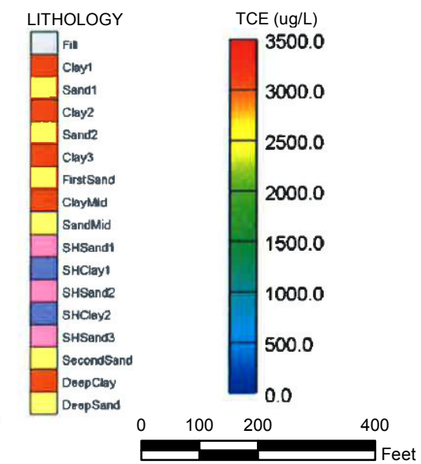


Figure 5

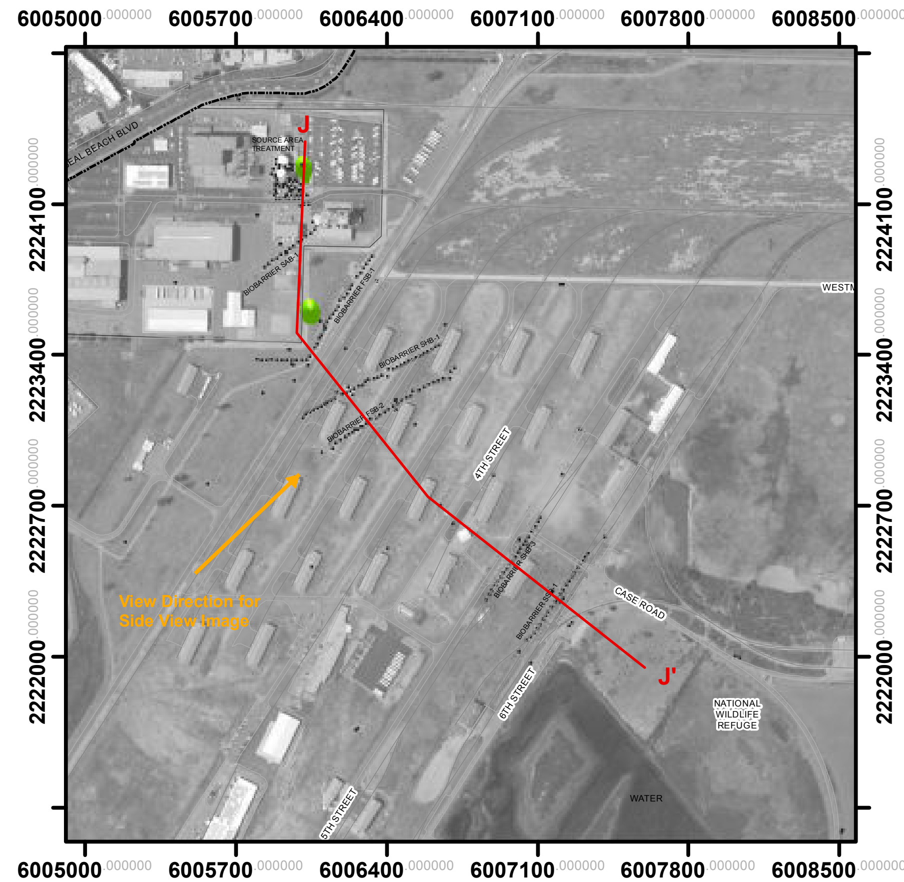
TRICHLOROETHYLENE EXTENT ABOVE 250 ug/L ALONG CROSS SECTION B-B', 2015 ANNUAL SAMPLING EVENT NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST SAN DIEGO, CALIFORNIA

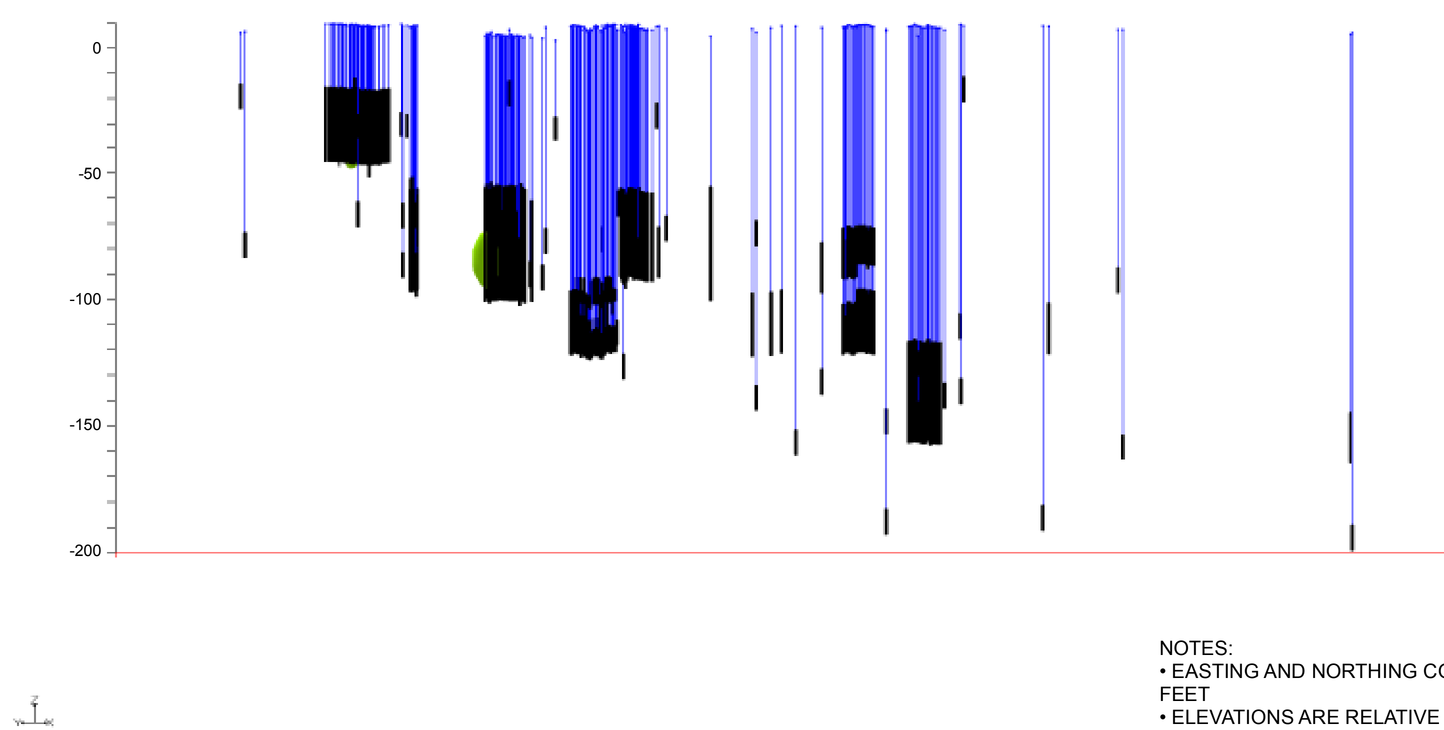
Sampling and Analysis Plan

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 5 lw.mxd



TCE: Iso-level = 250 ug/L, Volume = 8,231 cubic yards



Source: ECC-INSIGHT LLC Figure 3-4.mxd

NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X
- CROSS-SECTIONVIEW ANGLE TO THE NORTHEAST

GENERAL LOCATION MAP

WELL RISER

WELL SCREEN

PLUME SHELL

Figure 6

TRICHLOROETHENE EXTENT ABOVE 250 ug/L
2015 ANNUAL SAMPLING EVENT
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

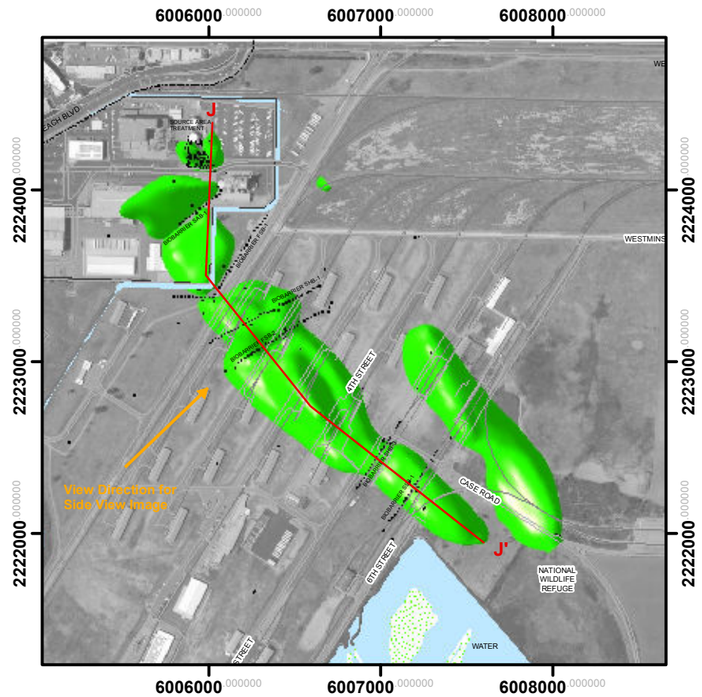
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

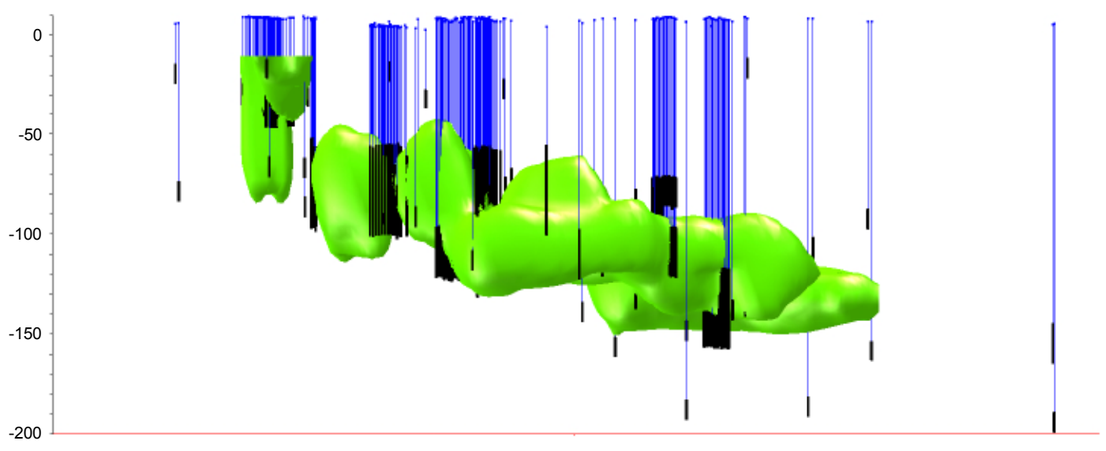
NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 7.mxd



1,2-DCE: Iso-level = 60 ug/L, Volume = 3,085,556 cubic yards



GENERAL LOCATION MAP

WELL RISER

WELL SCREEN

PLUME SHELL

NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X
- CROSS-SECTIONVIEW ANGLE TO THE NORTHEAST

Source: ECC-INSIGHT LLC Figure 3-5.mxd

Figure 7

CIS-1,2-DICHLOROETHENE EXTENT ABOVE 60 ug/L 2015 ANNUAL SAMPLING EVENT NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

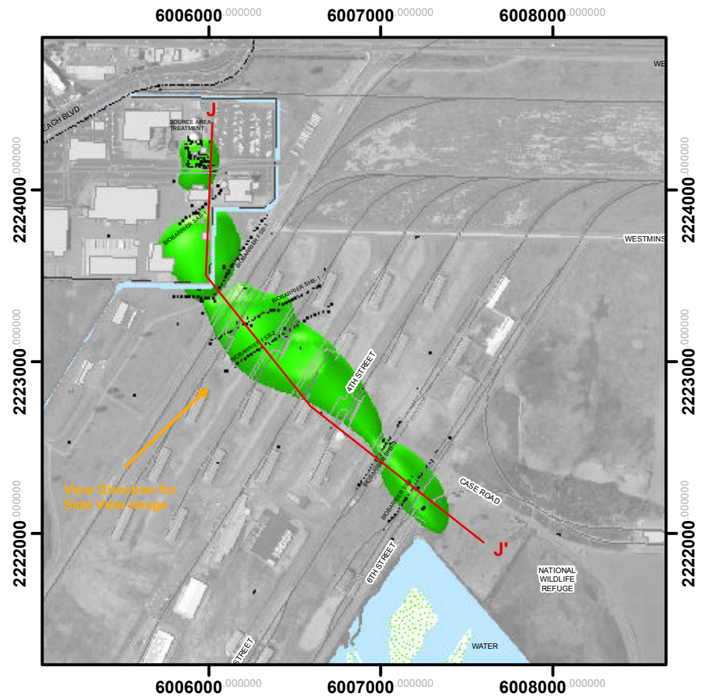
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

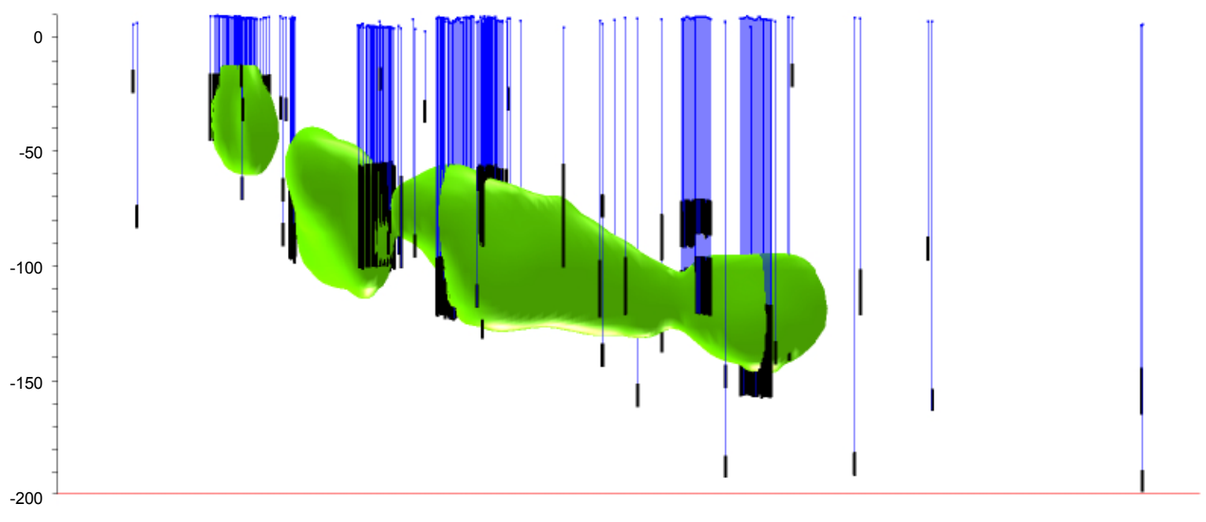
NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 7 LW.mxd



VINYL CHLORIDE: Iso-level = 20 ug/L, Volume = 1,154,815 cubic yards



NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X
- CROSS-SECTIONVIEW ANGLE TO THE NORTHEAST

Source: ECC-INSIGHT LLC Figure 3-6.mxd

GENERAL LOCATION MAP

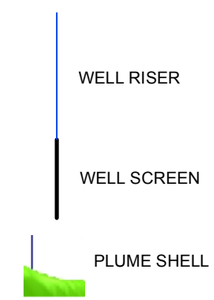


Figure 8

VINYL CHLORIDE EXTENT ABOVE 20 ug/L
2015 ANNUAL SAMPLING EVENT
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

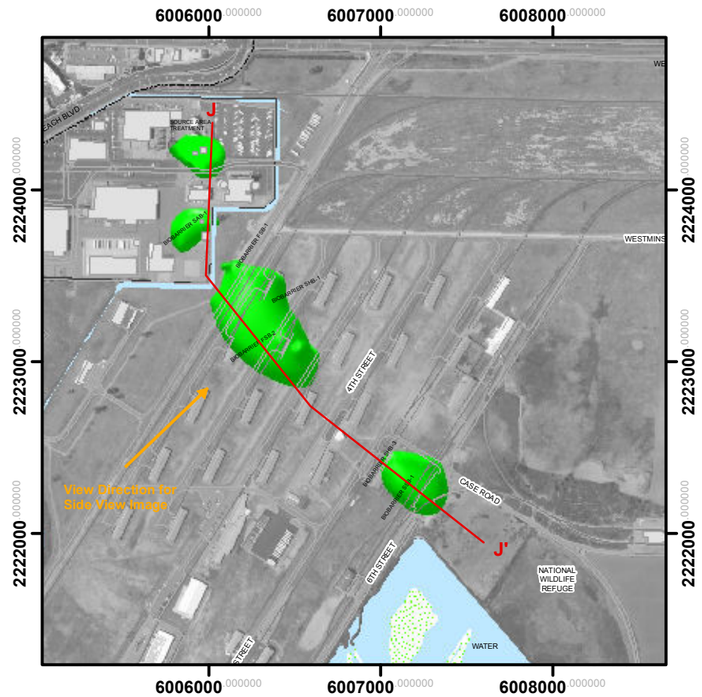
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

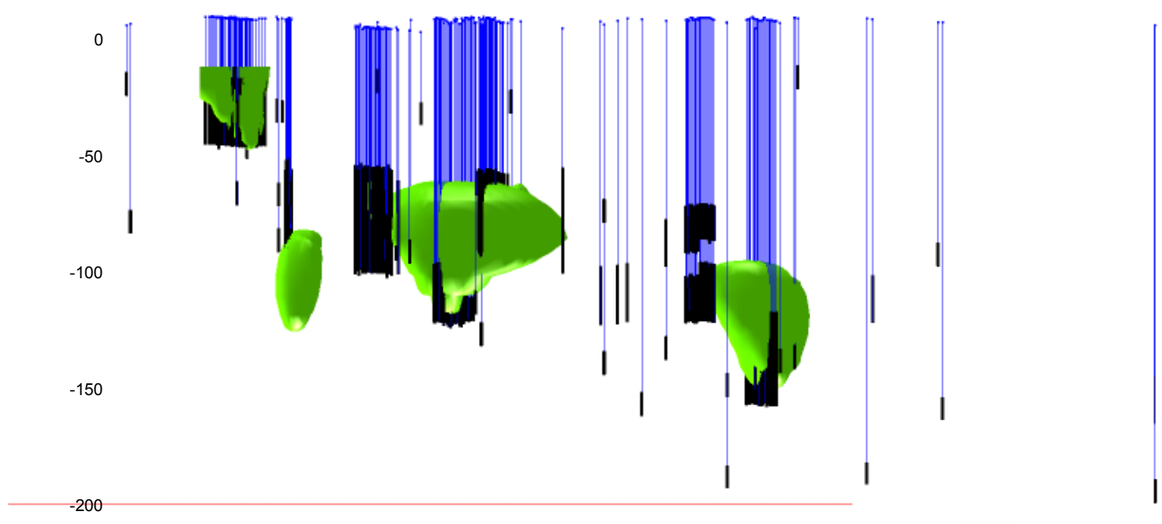
NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

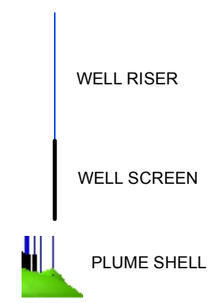
G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 8 LW.mxd



Ethene: Iso-level = 10 ug/L, Volume = 1,734,444 cubic yards



GENERAL LOCATION MAP



NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X
- CROSS-SECTIONVIEW ANGLE TO THE NORTHEAST

Source: ECC-INSIGHT LLC Figure 3-7.mxd

Figure 9

ETHENE EXTENT ABOVE 10 ug/L
2015 ANNUAL SAMPLING EVENT
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

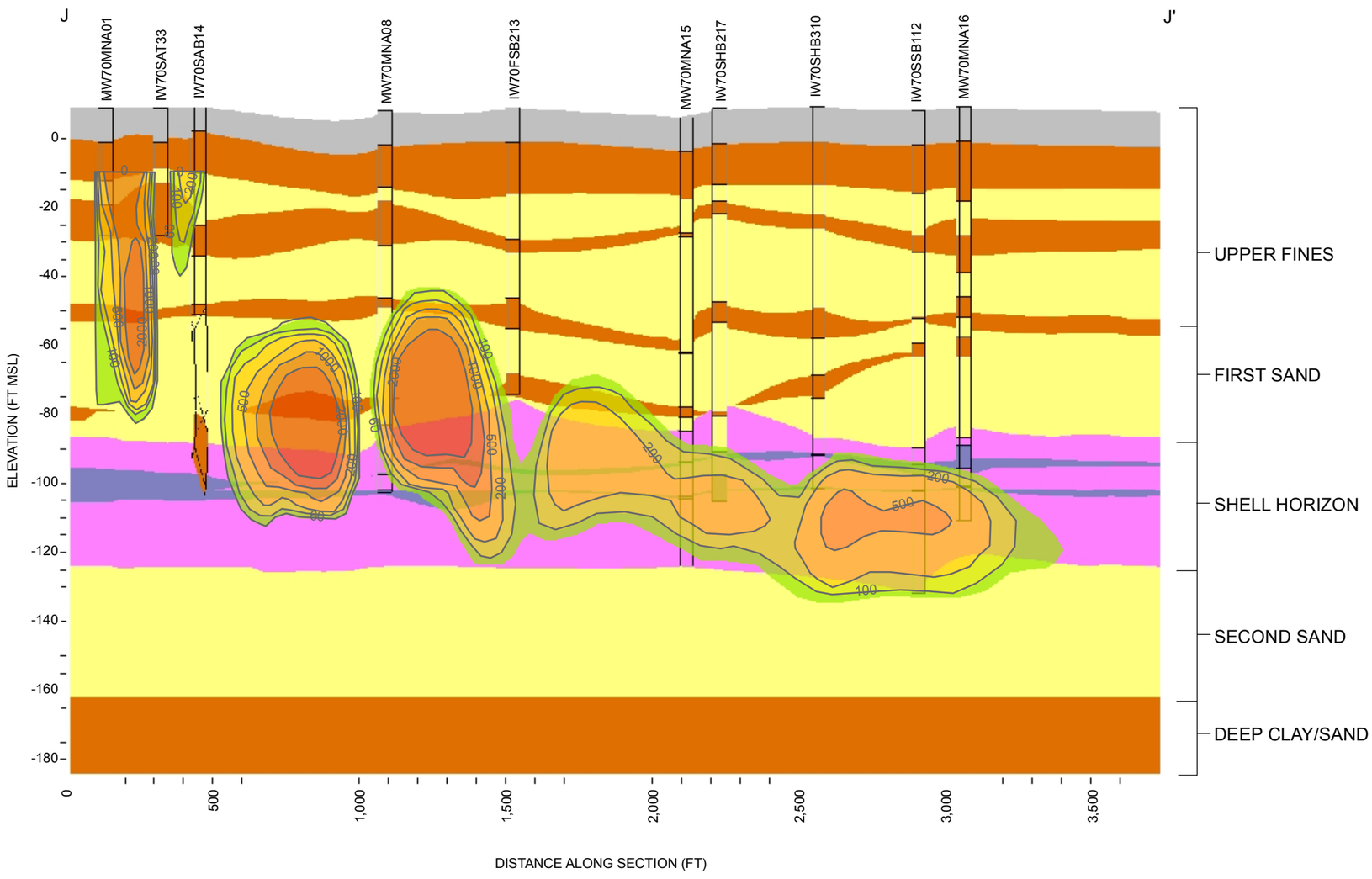
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

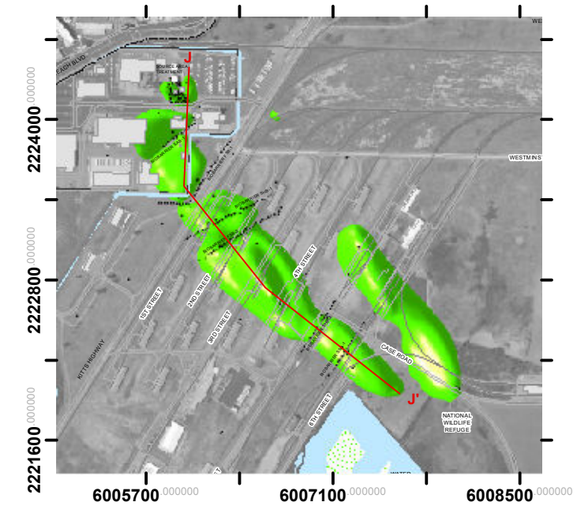
Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 9 LW.mxd





CROSS-SECTION LOCATION MAP

LITHOLOGY

- Fill
- Clay1
- Sand1
- Clay2
- Sand2
- Clay3
- FirstSand
- ClayMid
- SandMid
- SHSand1
- SHClay1
- SHSand2
- SHClay2
- SHSand3
- SecondSand
- DeepClay
- DeepSand

1,2-DCE (ug/L)

- 5500.0
- 2000.0
- 1000.0
- 500.0
- 60.0

0 250 500 1,000 Feet

NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X

Source: ECC-INSIGHT LLC Figure 3-8.mxd

Figure 10

CIS-1,2-DICHLOROETHENE EXTENT ABOVE 60 ug/L ALONG CROSSSECTION J-J', 2015 ANNUAL SAMPLING EVENT NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

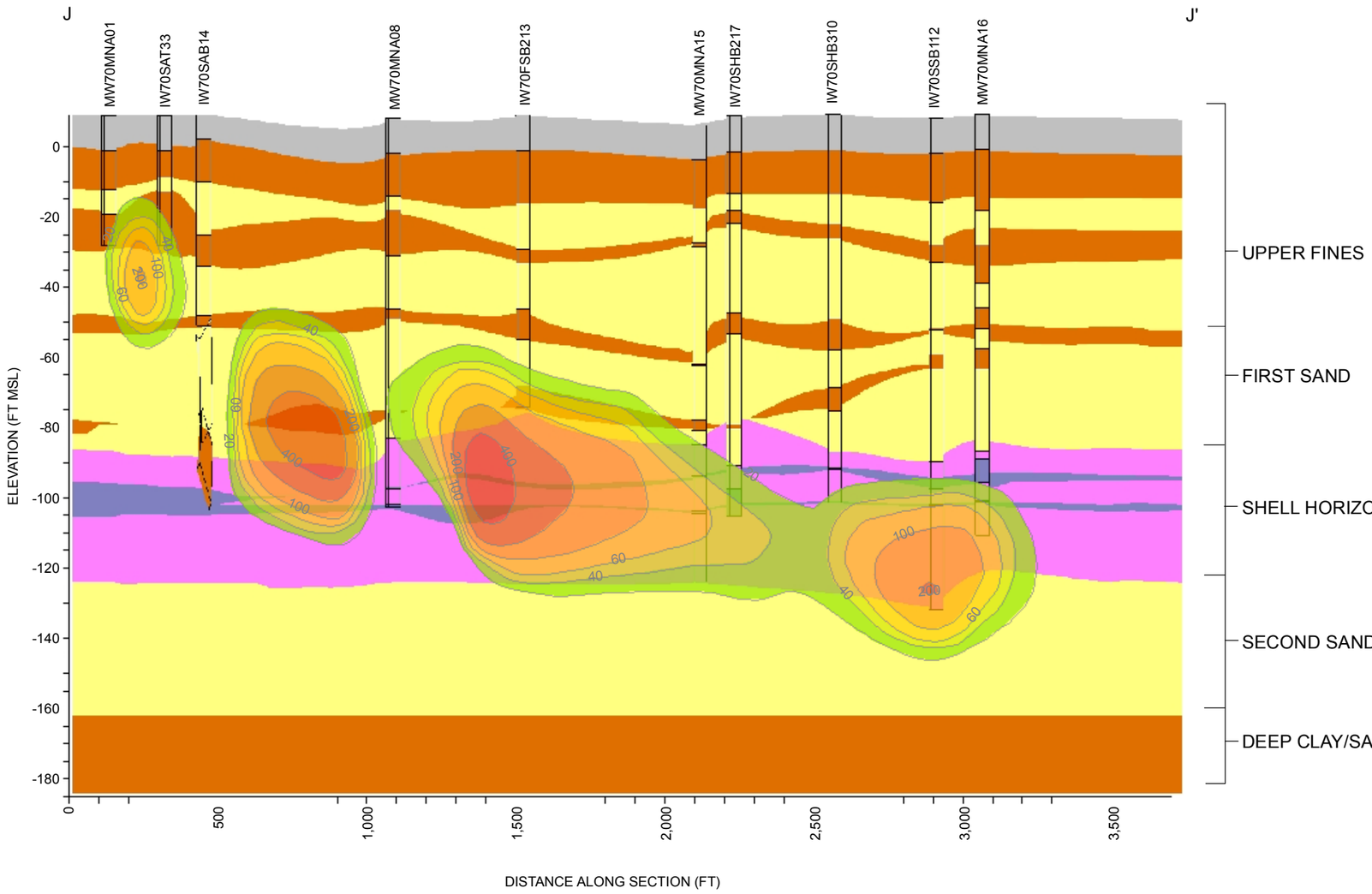
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

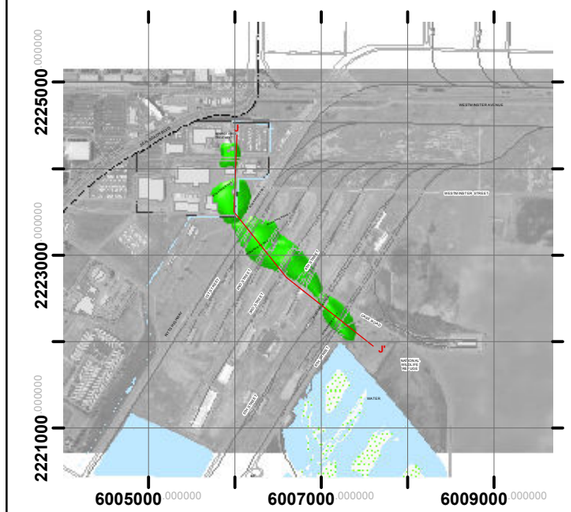
Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

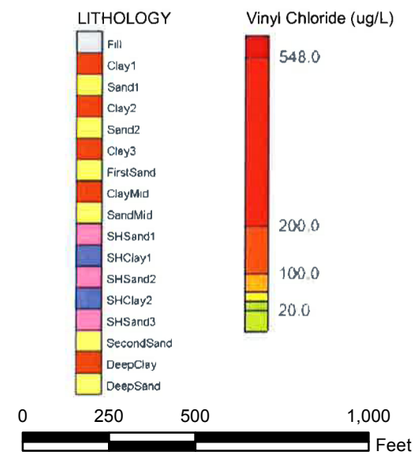
CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 10 LW.mxd





CROSS-SECTION LOCATION MAP



NOTES:
- EASTING AND NORTHING COORDINATES ARE IN NAD83 STATE PLANE, FEET
- ELEVATIONS ARE RELATIVE TO MEAN SEA LEVEL AS PER NGVD29 FEET
- CHEMISTRY UNITS ARE ug/L
- VERTICAL EXAGGERATION=10X

Source: ECC-INSIGHT LLC Figure 3-9.mxd

Figure 11

VINYL CHLORIDE EXTENT ABOVE 20 ug/L ALONG CROSSSECTION J-J', 2015 ANNUAL SAMPLING EVENT NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

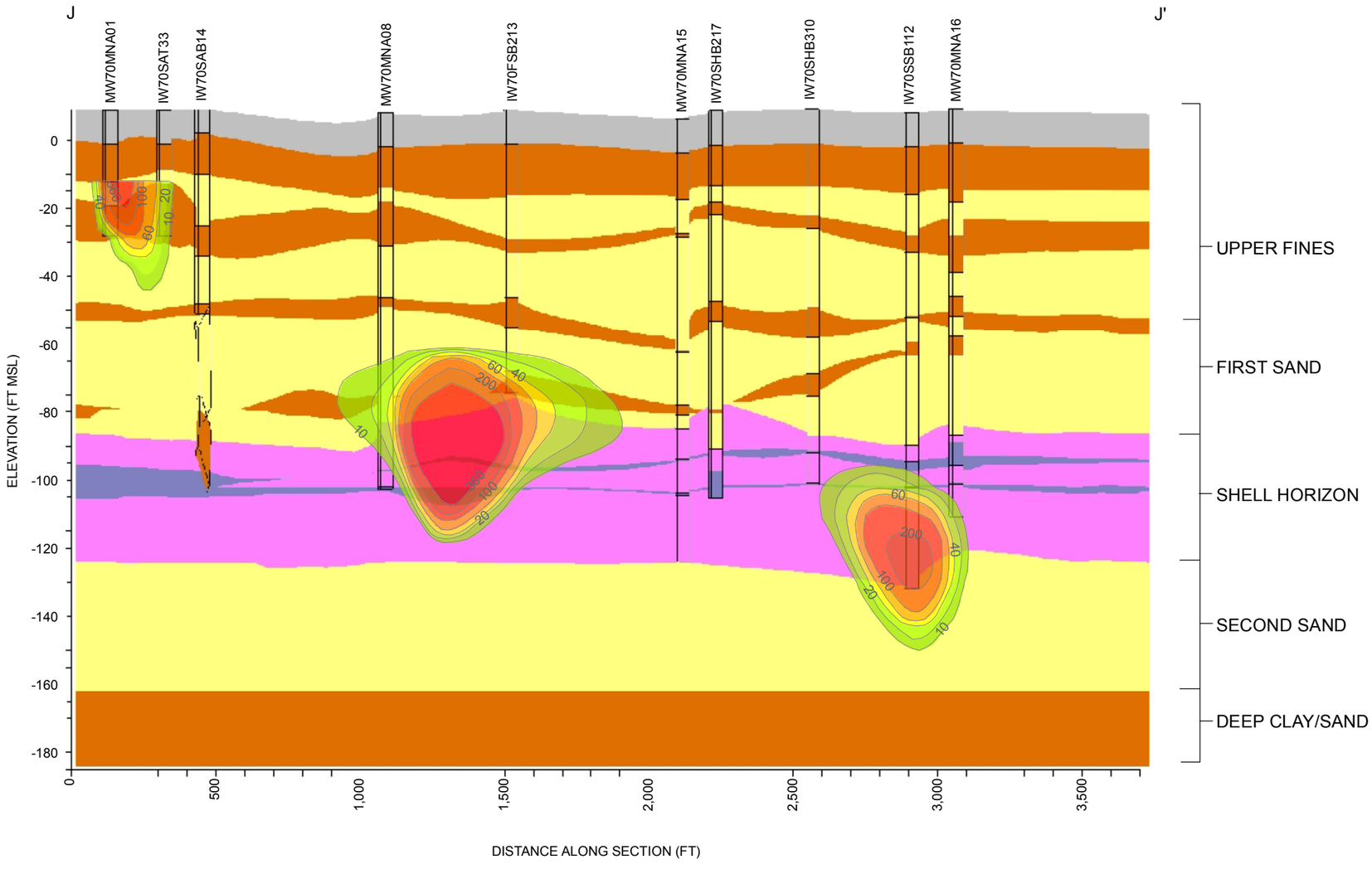
NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

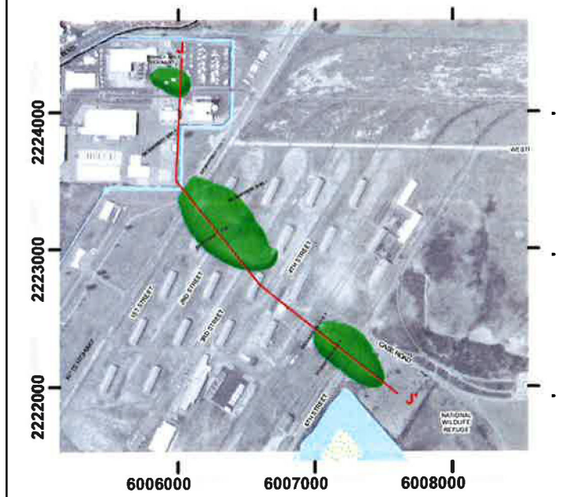
Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 11 LW.mxd





CROSS-SECTION LOCATION MAP

LITHOLOGY

- Fill
- Clay1
- Sand1
- Clay2
- Sand2
- Clay3
- FirstSand
- ClayMid
- SandMid
- SHSand1
- SHClay1
- SHSand2
- SHClay2
- SHSand3
- SecondSand
- DeepClay
- DeepSand

Ethene (ug/L)

- 360.0
- 200.0
- 100.0
- 40.0
- 10.0

0 250 500 1,000 Feet

Figure 12

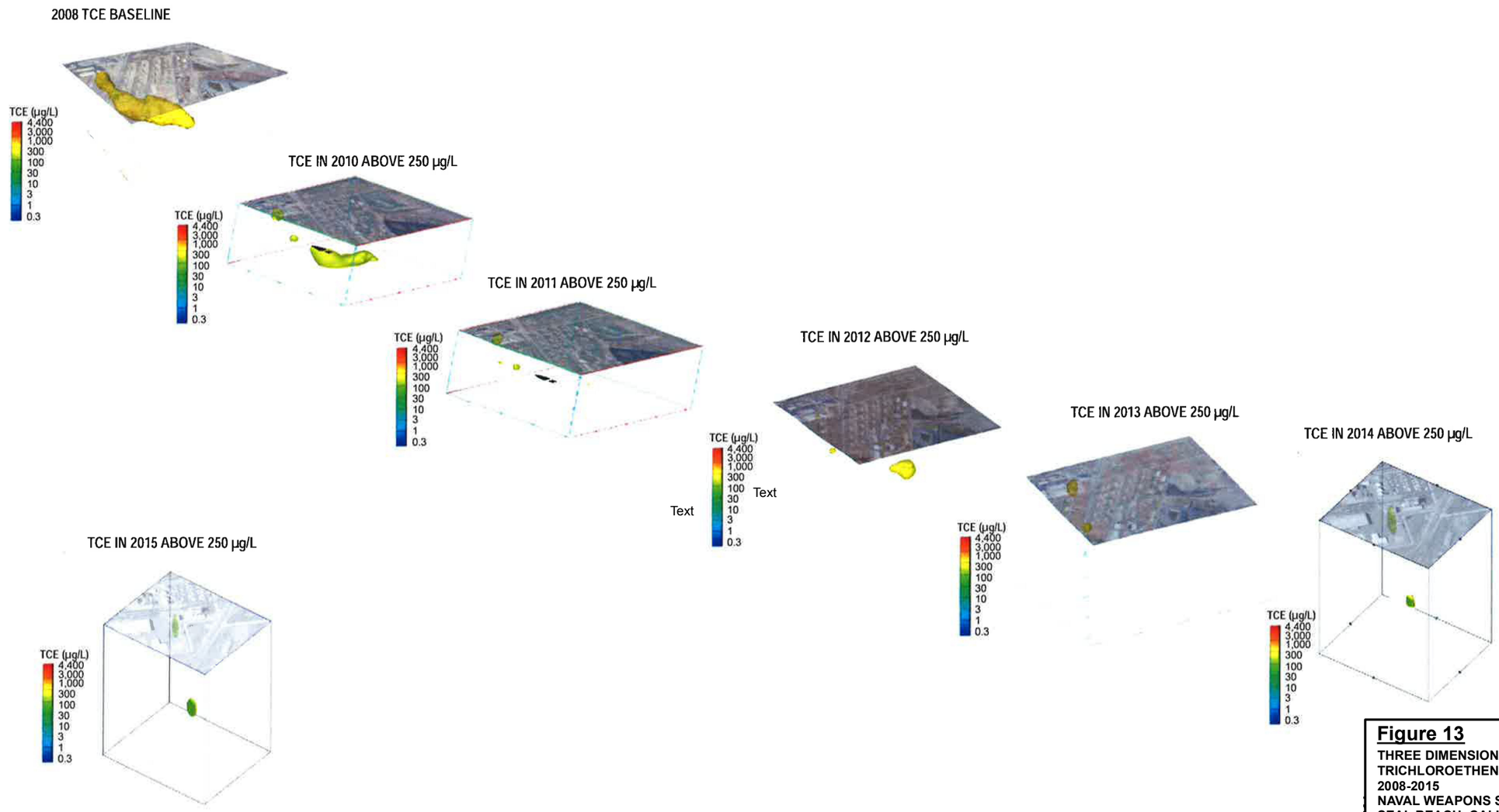
ETHENE EXTENT ABOVE 10 ug/L ALONG CROSSSECTION J-J',
2015 ANNUAL SAMPLING EVENT
NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

CB&I Federal Services LLC

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 13_B.mxd



Text

Text

Text

Figure 13

THREE DIMENSIONAL VIEWS OF TRICHLOROETHENE PLUME (>250 ug/L) 2008-2015

NAVAL WEAPONS STATION SEAL BEACH, SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND SOUTHWEST

SAN DIEGO, CALIFORNIA

Contract No. N62473-12-D-2005

Sampling and Analysis Plan

Text

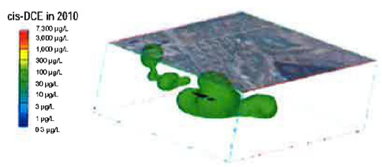
NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I Federal Services LLC

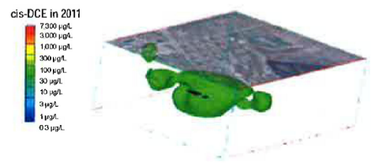
Source: ECC-INSIGHT LLC Figure 3-11.mxd

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 13 lw.mxd

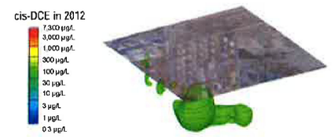
2010 CIS-DCE



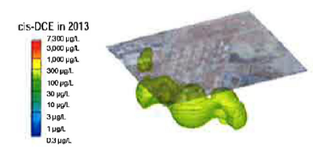
2011 CIS-DCE



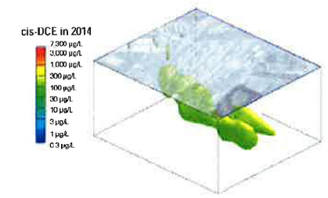
2012 CIS-DCE



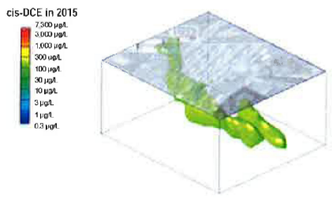
2013 CIS-DCE

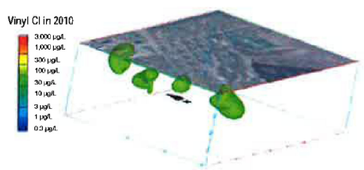


2014 CIS-DCE

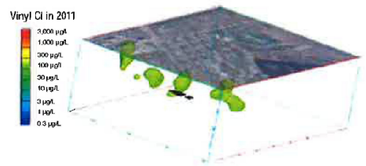


2015 CIS-DCE



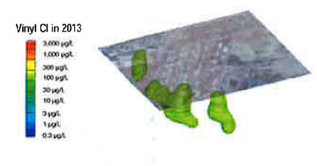


2010 VINYL CHLORIDE

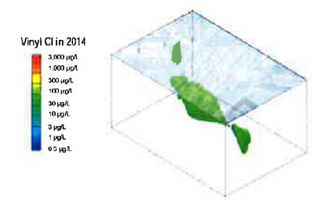


2011 VINYL CHLORIDE

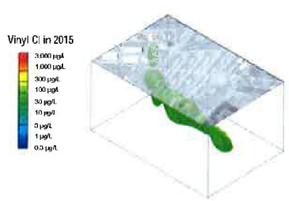
2012 VINYL CHLORIDE



2013 VINYL CHLORIDE



2014 VINYL CHLORIDE



2015 VINYL CHLORIDE

Figure 14

THREE DIMENSIONAL VIEWS OF
CIS-1,2-DICHLORETHENE PLUME (>60 ug/L)
AND VINYL CHLORIDE PLUME (>20 ug/L) 2010 TO 2015
NAVAL WEAPONS STATION SEAL BEACH,
SEAL BEACH, CALIFORNIA

NAVAL FACILITIES ENGINEERING COMMAND
SOUTHWEST
SAN DIEGO, CALIFORNIA
Contract No. N62473-12-D-2005

Sampling and Analysis Plan

NAVFAC Naval Facilities Engineering Command NAVFAC SOUTHWEST

CB&I **CB&I Federal Services LLC**

Source: ECC-INSIGHT LLC Figure 3-12.mxd

G:\NAVWPNSTA_Seal_Beach\Incoming_Data\From_ECC_Insight_Sept2016\SealBeach\MXD\2015_Annual_PMR_Sept2016\FIGURE 14 LW.mxd

APPENDIX A
FIELD FORMS

CHAIN OF CUSTODY

Lab ID Number:

Reference: IR Site 70, NAVWPNSTA Seal Beach - 500619 - 001

CB&I Federal Services, LLC

Company Name: *CB&I Federal Services, LLC*
Project Manager: ***Chris Johnson***
Phone/Fax Number: *714-669-7001 / 714-669-7060*
Send Report To: ***ATTN: Lynn Caragan***
Phone/Fax Number: *714-669-7022 / 714-669-7060*
Address: *420 Exchange, Suite 150*
City: *Irvine, CA 92602*
Project Contact: ***Lynn Caragan***
way-lynn.caragan@cbifederalservices.com

Project Number: *500619*
Project Name: *IR Site 70*
Project Location: *NAVWPNSTA Seal Beach*
Purchase Order: ***NA***
Lab Destination: ***EMAX***
Lab Contact: ***Richard Beauvil***
Address: *1835 W. 205th St.*
City: *Torrance, CA 90501*
Lab Phone #: *310-618-8889 x-118*

Location	Sample ID	Date	Time	Method Code	Matrix	No. of Containers	QC Level														MS/MSD - M

Bottle Code / Preservation Code

Number of Containers

Requesting Testing Program

Turnaround Time	Sampler(s) Name(s)
Std.	
Rush:	

Special Instructions:

Method Codes

C = Composite
G = Grab
LF - Low Flow

Condition upon Receipt

Cooler temp (°C):
Received on ice: Y N
Preserved: Y N
Correct container(s): Y N
Seal(s) intact: Y N
Tracking #:

Associated Forms

Analyte list: Y N
Sampling Logbook Copy: Y N
Other: Y N

Matrix Codes

SO =Soil
GW = Ground Water
SL = Sludge
WW = Waste Water
A = Air Sample
SOL = Other Solid

Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Print Name and Company:			Print Name and Company:		
Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Print Name and Company:			Print Name and Company:		
Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Print Name and Company:			Print Name and Company:		

Bottle Codes

A = 120ml, 250ml, 500ml, or 1-L HDPE
B = 40-ml VOA Vial
C = 1-L Glass Amber
D = 4 or 8 oz glass jar
E = Encores

Preservative Codes

1 = Hydrochloric Acid (HCl)
2 = Nitric Acid (HNO3)
3 =Zn actate, NaOH
4 = Ice
5 = None

PAGE	1 OF 1
DATE	

SAMPLE COLLECTION LOG

Project Name: IR Site 70 RAO Well ID: _____

Project Number: 500619 Sample Collected By: _____

Project Location: NAVWPNSTA Seal Beach

Purging Method/Equipment: Micropurge

Sampling Equipment: Non-dedicated QED Portable Bladder Pump /dedicated tubing

Flow Rate During Purging : 300 - 500 ml/min with water level drawdown less than 0.3 feet

Note: Flowrate for VOC sampling should be no greater than 100 ml/min

Daily field instrument calibration verification performed? Yes ___ No ___

Pump Start Time: _____

Measuring Point Top of Casing Drawdown depth limit (DDL) = DTWi + 0.3 (ft)

Casing ID (in.) _____ depth of pump inlet (ft): _____

Depth to Water - Initial (DTWi) (ft) _____

Depth to Well Bottom (ft) _____

Screen Interval (ft): _____

FIELD CONDITIONS

Weather Conditions: ☐ Sunny ☐ Rain ☐ Wind Temperature: _____

PURGING INFORMATION

Time	Purge Rate (ml/min)	Depth to Water (DTW) (feet)	Specific Conductance (mS or µS /cm)	pH pH Units	Temp (˚C / F)	Turbidity (FNU/NTU)	ORP mV	DO (µg or mg/L)
Stabilization criteria:		drawdown limit 0.3 ft	± 10%	± 0.1	± 1	No Criteria	± 10 mV	± 10%

SAMPLING INFORMATION

Sample Number:		
Sample Date/Time:	/	
QC Sample Collected:	___ MS/MSD	_____ Field Duplicate Collected
Analyses / Bottles:	VOC (8260B) - 3 VOA - HCl ☐	☐
	Dissolved Gases - 3 VOA, HCl ☐	
	Anions - Poly no Preservative ☐	
	TOC-8 oz glas, H2SO4 ☐	
	Sulfide-150 ml-HDPE NaOH/ ZnAoc2 ☐	

Notes:

SOIL VAPOR SAMPLE COLLECTION LOG

Project Name: IR Site 70 RAO

Site Location: NAVWPNSTA Seal Beach

Project No.: 500619

Well ID: _____

Sample No.: _____

Date and Time Collected: _____

Sample Collected by: _____

EQUIPMENT

Purging Method/Equipment:

Purge Volume :

Flow Rate During Sampling: <200 ml/min. Use flow control regulator from lab (for soil gas wells)

Summa Canister ID # _____

Summa Canister Initial Pressure (in/Hg) _____

Summa Canister Final Pressure (in/Hg) _____

Flow Contoller ID # _____

Flow Contoller Rate (ml/min) _____

Sample Start Time: _____

Sample End Time: _____

FIELD CONDITIONS

Wind Direction (circle one): None N S E W NE NW SE SW Ambient Air Temp. (^{o}F):

Weather Conditions: _____

Notes:

PID READING = ppm

Leak check compound = IPA

SAMPLE PACKAGING

Container(s) Type and Volume	No. of Containers	Preservatives	Parameters
Summa Canister	1	None	VOCs - TO-15A

APPENDIX B
LABORATORY CERTIFICATIONS
(provided on electronic copy only)

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

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**

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-Cl- 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

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

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

Field of Testing: 120 - Physical Properties of Hazardous Waste

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

Certificate of Accreditation

ISO/IEC 17025:2005 Certificate Number L2278

EMAX Laboratories, Inc.

1835 W. 205th 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 9th 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

LABORATORY ACCREDITATION BUREAU

Certificate # L2278

Scope of Accreditation
For
EMAX Laboratories, Inc.

1835 W 205th 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
Gravimetric	EPA 160.5	Settleable 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

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 200.7	Iron
ICP	EPA 200.7	Lead
ICP	EPA 200.7	Lithium
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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	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
Titrimetric	EPA 377.1	Sulfite
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC	EPA 608	beta-BHC
GC	EPA 608	delta-BHC
GC	EPA 608	gamma-BHC (Lindane)
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-MS	EPA 624	Chlorobenzene
GC-MS	EPA 624	2-Chloroethyl vinyl ether
GC-MS	EPA 624	Chloroethane
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-Trichloro1,2,2-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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-MS	EPA 625	Benzo(e)pyrene
GC-MS	EPA 625	Benzo(g,h,i)perylene
GC-MS	EPA 625	Benzo(k)fluoranthene
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-MS	EPA 625	Hexachloroethane
GC-MS	EPA 625	Indeno(1,2,3-cd)pyrene
GC-MS	EPA 625	Isophorone
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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 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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC	EPA 8082 / 8082A	PCB 110
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	Total 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

Non-Potable Water		
Technology	**Method**	**Analyte**
GC	EPA 8151A	4-Nitrophenol
GC	EPA 8151A	Pentachlorophenol
GC	EPA 8151A	Picloram
GC	EPA 8151A	Silvex
GC	EPA 8151A	2,4,5-T
GC-MS	EPA 8260B / 8260C	Acetone
GC-MS	EPA 8260B / 8260C	Acetonitrile
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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-Trichloro1,2,2-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	Allyl Chloride

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-MS	EPA 8260B / 8260C	Benzyl chloride
GC-MS	EPA 8260B / 8260C	Chloroprene
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
GC-MS	EPA 8270C / 8270D	3/4-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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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 (18th)	Ammonia
Spectrometric	SM 4500-NH3F	Ammonia
Spectrometric	SM 4500-NOrgC NH3F	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
Electrical Conductivity	SM 2520B	Salinity
Gravimetric	SM 2540C	TDS
Gravimetric	SM 2540D	TSS
Gravimetric	SM 2540B	Total Residue
Gravimetric	SM 2540F	Settleable Residue
Spectrometric	SM 3500-FeB	Ferrous iron
IC	SM 4110B	Bromate
IC	SM 4110B	Bromide
IC	SM 4110B	Chloride

Non-Potable Water		
Technology	**Method**	**Analyte**
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
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
Preparation	**Method**	**Type**
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

Drinking Water		
Technology	**Method**	**Analyte**
Colorimetric	EPA 110.2	Color
Platinum Electrode	EPA 120.1	Specific Conductance
Electrode	EPA 150.1	pH
Gravimetric	EPA 160.1	TDS
Gravimetric	EPA 160.2	TSS

Drinking Water		
Technology	**Method**	**Analyte**
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
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

Drinking Water		
Technology	**Method**	**Analyte**
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
Titrimetric	EPA 330.3	Residual Chlorine
Spectrometric	EPA 335.2	Cyanide
Spectrometric	EPA 350.2	Ammonia
Spectrometric	EPA 351.3	TKN
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 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
Spectrometric	EPA 425.1	MBAS
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

Drinking Water		
Technology	**Method**	**Analyte**
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
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

Drinking Water		
Technology	**Method**	**Analyte**
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-Trichloro1,2,2-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
HPLC-MS	EPA 6850	Perchlorate
Colorimetric	SM 2120B	Color
Threshold Odor Test	SM 2150B	Odor
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
Titrimetric	SM 4500-ClB	Residual Chlorine
Titrimetric	SM 4500-Cl-B	Chloride
Spectrometric	SM 4500-CNE	Cyanide
Electrode	SM 4500 HB	pH
Spectrometric	SM 4500-NH3C (18th)	Ammonia
Spectrometric	SM 4500-NH3F	Ammonia
Spectrometric	SM 4500-NO2B	Nitrite-N
Spectrometric	SM 4500-NO3E	Nitrate-N
Spectrometric	SM 4500-NOrgC NH3F	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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
Visual	s.7.3 SW-846	Reactive Cyanide
Visual	s.7.3 SW-846	Reactive Sulfide
Spectrometric	SM4500-NH3C (18th)	Ammonia
Spectrometric	SM4500-NH3F	Ammonia
Spectrometric	SM4500-NOrgC NH3F	TKN
Spectrometric	SM4500-NO2B	Nitrite-N
Spectrometric	SM4500-NO3E	Nitrate-N
Spectrometric	SM4500PE	Ortho-phosphate
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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)
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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 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	Total Demeton

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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-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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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-Trichloro1,2,2-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
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	Allyl Chloride
GC-MS	EPA 8260B / 8260C	Benzyl chloride
GC-MS	EPA 8260B / 8260C	Chloroprene
GC-MS	EPA 8260B / 8260C	Methyl Acetate
GC-MS	EPA 8260B / 8260C	Methylcyclohexane
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
GC-MS	EPA 8270C / 8270D	3/4-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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
HPLC	EPA 8332	Nitroglycerine
HPLC	EPA 8332	PETN
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

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
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
Spectrometric	EPA 9014	Amenable Cyanide
Gravimetric	EPA 9071B	Oil & Grease
GFAA	CA 939M	Organo Lead
Preparation	**Method**	**Type**
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
SPLP	EPA 1312	Leaching
Floricil 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

Air and Emissions		
Technology	**Method**	**Analyte**
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

Air and Emissions		
Technology	**Method**	**Analyte**
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
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

Air and Emissions		
Technology	**Method**	**Analyte**
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: _____ Date: December 7, 2015

R. Douglas Leonard
Chief Technical Officer

Reissued: 1/9/14 Revised: 9/25/14 Revised: 12/7/15

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

TestAmerica Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

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.: **2897**

Expiration Date: **1/31/2018**

Effective Date: **2/1/2016**

Sacramento, California
subject to forfeiture or revocation

Christine Sotelo, Chief
Environmental Laboratory Accreditation Program

CALIFORNIA STATE
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

TestAmerica Sacramento

880 Riverside Parkway
West Sacramento, CA 95605
Phone: (916) 373-5600

Certificate No. 2897
Expiration Date 1/31/2018

Field of Testing: 102 - Inorganic Chemistry of Drinking Water

102.045	001	Perchlorate	EPA 314.0	
102.047	001	Perchlorate	EPA 331.0	

Field of Testing: 105 - Semi-volatile Organic Chemistry of Drinking Water

105.230	001	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	EPA 1613B	

Field of Testing: 108 - Inorganic Chemistry of Wastewater

108.020	001	Conductivity	EPA 120.1	
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	006	Silica, Dissolved	EPA 200.7	
108.112	007	Sodium	EPA 200.7	
108.112	008	Phosphorus, Total	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	
108.232	003	Nitrate-Nitrite (as N)	EPA 353.2	
108.323	001	Chemical Oxygen Demand	EPA 410.4	
108.410	001	Alkalinity	SM2320B-1997	
108.420	001	Hardness (calculation)	SM2340B	
108.430	001	Conductivity	SM2510B	
108.440	001	Residue, Total	SM2540B	
108.441	001	Residue, Filterable TDS	SM2540C	
108.442	001	Residue, Non-filterable TSS	SM2540D	
108.490	001	Hydrogen Ion (pH)	SM4500-H+ B	
108.528	001	Nitrate-Nitrite (as N)	SM4500-NO3- E-2000	Interim

As of 2/2/2016 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

108.528	002	Nitrite (as N)	SM4500-NO3- E-2000	Interim
108.528	003	Nitrate (as N)	SM4500-NO3- E-2000	Interim

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	020	Gold	EPA 200.8
109.020	021	Iron	EPA 200.8
109.020	022	Tin	EPA 200.8
109.020	023	Titanium	EPA 200.8

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

110.040	000	Purgeable Organic Compounds	EPA 624

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

111.100	000	Acid/base/neutral Organic Compounds	EPA 625
111.103	000	Nitrosamines	EPA 625
111.111	000	Dioxins and Dibenzofurans	EPA 1613B

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
114.140	001	Mercury	EPA 7470A
114.141	001	Mercury	EPA 7471A
114.240	001	Corrosivity - pH Determination	EPA 9040B
114.241	001	Corrosivity - pH Determination	EPA 9045C
114.250	001	Fluoride	EPA 9056

Field of Testing: 115 - Extraction Test of Hazardous Waste

115.021	001	TCLP Inorganics	EPA 1311	Interim
115.022	001	TCLP Extractables	EPA 1311	Interim
115.030	001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II	
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

116.080	000	Volatile Organic Compounds	EPA 8260B	
116.080	000	Volatile Organic Compounds	EPA 8260B	
116.080	120	Oxygenates	EPA 8260B	
116.080	120	Oxygenates	EPA 8260B	
116.100	001	Total Petroleum Hydrocarbons - Gasoline	LUFT GC/MS	
116.100	001	Total Petroleum Hydrocarbons - Gasoline	LUFT GC/MS	
116.100	002	Benzene	LUFT GC/MS	
116.100	003	Toluene	LUFT GC/MS	
116.100	004	Xylenes	LUFT GC/MS	
116.100	005	Methyl tert-butyl Ether (MTBE)	LUFT GC/MS	
116.100	010	BTEX and MTBE	LUFT GC/MS	
116.100	010	BTEX and MTBE	LUFT GC/MS	
116.110	001	Total Petroleum Hydrocarbons - Gasoline	LUFT	Interim

Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B	
117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015B	
117.016	001	Diesel-range Total Petroleum Hydrocarbons	LUFT	
117.016	001	Diesel-range Total Petroleum Hydrocarbons	LUFT	
117.110	000	Extractable Organics	EPA 8270C	
117.110	000	Extractable Organics	EPA 8270C	
117.120	000	Dioxins and Dibenzofurans	EPA 8280A	
117.120	000	Dioxins and Dibenzofurans	EPA 8280A	
117.130	000	Dioxins and Dibenzofurans	EPA 8290	
117.130	000	Dioxins and Dibenzofurans	EPA 8290	
117.170	000	Nitroaromatics and Nitramines	EPA 8330	
117.170	000	Nitroaromatics and Nitramines	EPA 8330	Interim
117.171	000	Nitroaromatics and Nitramines	EPA 8330A	
117.210	000	Pesticides	EPA 8081A	
117.210	000	Pesticides	EPA 8081A	
117.220	000	PCBs	EPA 8082	
117.220	000	PCBs	EPA 8082	

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA LABORATORIES SACRAMENTO
880 Riverside Parkway
West Sacramento, CA 95605
Lisa Stafford Phone: 916 374 4308

ENVIRONMENTAL

Valid To: January 31, 2017 Certificate Number: 2928.01

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 2009 TNI Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.0 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

Inductively Coupled Plasma (ICP), ICP-Mass Spectroscopy, Atomic Absorption Spectroscopy (flame), Gas Chromatography(GC), GC- Mass Spectroscopy, High Resolution Gas Chromatography/High Resolution Mass Spectroscopy, Liquid Chromatography(LC),LC- Mass Spectroscopy, Ion Chromatography, Spectrophotometry, Misc.-Electronic Probes

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Metals			
Aluminum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Antimony	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Arsenic	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Barium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Beryllium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Boron	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	----------
Cadmium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Calcium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Chromium (Total)	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Chromium (Hexavalent)	EPA 7196A	EPA 7196A	----------
Cobalt	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Copper	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Iron	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Lead	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Magnesium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Manganese	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Mercury	EPA 7470A	EPA 7471A/7471B	----------
Molybdenum	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Nickel	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Phosphorus	EPA 6020/6020A	EPA 6020/6020A	----------
Potassium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Selenium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Silica	EPA 6010B/6010C	----------	----------
Silicon	EPA 6010B/6010C	----------	----------
Silver	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Sodium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Strontium	EPA 6020/6020A	EPA 6020/6020A	----------
Thallium	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	EPA 6020/6020A
Tin	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	----------
Titanium	EPA 6010B/6020/6010C/6020A	EPA 6010B/6020/6010C/6020A	----------
Uranium	EPA 6020/6020A	EPA 6020/6020A	----------
Vanadium	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Zinc	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A	EPA 6020/6020A
Nutrients			
Nitrate	EPA 353.2/9056A/300.0	EPA 353.2/ 9056A/300.0	----------
Nitrate-nitrite	EPA 353.2	EPA 353.2	----------
Nitrite	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	----------
Orthophosphate	EPA 9056A/300.0	EPA 9056A/300.0	----------
Wet Chemistry			
Alkalinity	SM 2320B (1997)	----------	----------
Chemical Oxygen Demand	EPA 410.4	----------	----------
Nitrocellulose	WS-WC-0050/353.2 Modified	WS-WC-0050/353.2 Modified	----------
Perchlorate	EPA 6850	EPA 6850	----------
pH	EPA 9040B/9040C	EPA 9045C/9045D	----------
Bromide	EPA 9056A/300.0	EPA 9056A/300.0	----------
Chloride	EPA 9056A/300.0	EPA 9056A/300.0	----------
Fluoride	EPA 9056A/300.0	EPA 9056A/300.0	----------
Sulfate	EPA 9056A/300.0	EPA 9056A/300.0	----------
Solids, Total	SM2540B (1997)	----------	----------
Solids, Total Suspended	SM2540D (1997)	----------	----------
Solids, Total Dissolved	SM2540C (1997)	----------	----------
%Moisture	----------	ASTM D2216	
TSP (Total Suspended Particulate)	----------	----------	40CFR Part 50 App B
PM10	----------	----------	40CFR Part 50 App J

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Hazardous Waste Characteristics			
TCLP Extractables	----------	EPA 1311	----------
TCLP Inorganics	----------	EPA 1311	----------
Purgeable Organics (volatiles)			
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	----------
1,1,1-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1,2-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-2-Trichloro-1,2-2-trifluorethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15-SIM
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,1-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	----------
1,2,3-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2,3-Trichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2,4-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2,4-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C	EPA 8260B/8260C	----------
1,2-Dibromoethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,3,5-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
1,3-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,3-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	----------
1,4-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
1,4-Dioxane	----------	----------	TO14A/TO15/ TO15 SIM
1-Chlorocyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	----------
2,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	----------
2-Butanone (MEK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
2-Hexanone (MBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
2-Methyl-2-propanol (tert-Butyl Alcohol, TBA)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
4-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	----------
4-Ethyltoluene	----------	----------	TO14A/TO15
4-Isopropyltoluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
4-Methyl-2-pentanone (MIBK)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acetone	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Acrolein	----------	----------	TO14A/TO15/ TO15 SIM
Allyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Alpha Methyl Styrene	----------	----------	TO14A/TO15
Benzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Benzyl Chloride	----------	----------	TO14A/TO15/ TO15 SIM
Bromobenzene	EPA 8260B/8260C	EPA 8260B/8260C	----------
Bromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	----------
Bromodichloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Bromoform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Bromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Butadiene (1,3-Butadiene)	----------	----------	TO14A/TO15/ TO15 SIM
Butane	----------	----------	TO14A/TO15
Carbon Disulfide	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Carbon Tetrachloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chlorodifluoromethane	----------	----------	TO14A/TO15
Chloroethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chloroform	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Chloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
cis-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
cis-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Cyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dibromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Dibromomethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Dichlorodifluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Diisopropyl Ether (DIPE)	EPA 8260B/8260C	EPA 8260B/8260C	----------
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	EPA 15/16

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Ethyl Acetate	----------	----------	TO14A/TO15
Ethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Ethylmethacrylate	EPA 8260B/8260C	EPA 8260B/8260C	----------
Ethyl tert-butyl Ether (ETBE)	EPA 8260B/8260C	EPA 8260B/8260C	----------
Hexachlorobutadiene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Hexane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Iodomethane	EPA 8260B/8260C	EPA 8260B/8260C	----------
Isobutanol (2-Methyl-1-propanol)	EPA 8260B/8260C	EPA 8260B/8260C	----------
Isooctane (2,2,4-Trimethylpentane)	----------	----------	TO14A/TO15
Isopropyl Alcohol	----------	----------	TO14A/TO15
Isopropylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
m & p Xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Methyl tert-butyl Ether (MTBE)	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Methylene Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Naphthalene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
n-Butanol	----------	----------	TO14A/TO15
n-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
n-Heptane	----------	----------	TO14A/TO15
n-Nonane	----------	----------	TO14A/TO15
n-Octane	----------	----------	TO14A/TO15
n-Propylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
o-Xylene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Pentane	----------	----------	TO14A/TO15
Propene	----------	----------	TO14A/TO15
sec-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Styrene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
t-Amyl methyl Ether (TAME)	EPA 8260B/8260C	EPA 8260B/8260C	----------
t-1,4-Dichloro-2-Butene	EPA 8260B/8260C	EPA 8260B/8260C	----------
tert-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Tetrachloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Tetrahydrofuran	----------	----------	TO14A/TO15
Toluene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
trans-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
trans-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Trichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Trichlorofluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Vinyl Acetate	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15
Vinyl Bromide	----------	----------	TO14A/TO15
Vinyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Xylenes, Total	EPA 8260B/8260C	EPA 8260B/8260C	TO14A/TO15/ TO15 SIM
Carbon Dioxide	----------	----------	ASTM1946D/3C
Nitrogen	----------	----------	ASTM1946D/3C
Oxygen	----------	----------	ASTM1946D/3C
Helium	----------	----------	ASTM1946D/3C
Hydrogen	----------	----------	ASTM1946D/3C
Methane	----------	----------	ASTM1946D/3C
Gasoline Range Organics (GRO)	EPA 8260B/AK101MS	EPA 8260B/AK101MS	TO14A/TO15
TPH as Gasoline	EPA 8260B/AK101MS	EPA 8260B/AK101MS	TO14A/TO15
Extractable Organics (semivolatiles)			
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	----------
1,2,4-Trichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,2-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,2-Diphenylhydrazine (as Azobenzene)	EPA 8270C/8270D	EPA 8270C/8270D	----------
1,3-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,3-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1,4-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
1-Methylnaphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,4-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2,6-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Methylnaphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
2-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3&4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4,6-Dinitro-2-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Acenaphthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Acenaphthylene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Aniline	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(a)anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(a)pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(b)fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(g,h,i)perylene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzo(k)fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Benzoic Acid	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Benzyl Alcohol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Benzyl butyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Biphenyl	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroethoxy) Methane	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroethyl) Ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Bis(2-chloroisopropyl) Ether	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Chrysene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
			TO-13A Modified
Bis (2-ethylhexyl) Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Dibenz(a,h)anthracene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Diethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Dimethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Di-n-butyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Di-n-octyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Fluoranthene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Fluorene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachlorobutadiene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Indeno(1,2,3-c,d) Pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Naphthalene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Nitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
n-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Pentachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Phenanthrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM/	WS-MS-0006/ TO-13A/ TO-13A Modified
Phenol	EPA 8270C/8270D	EPA 8270C/8270D	TO-13A
Pyrene	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	EPA 8270C/8270D/8270C-SIM/ 8270D-SIM	WS-MS-0006/ TO-13A/ TO-13A Modified
Pyridine	WS-MS-0005	WS-MS-0005	----------
1,4-Dioxane	WS-MS-0011/8270C-SIM Modified	WS-MS-0011/8270C-SIM Modified	----------
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D/ AK102	EPA 8015B/8015C8015D/ AK102	----------
Residual Range Organics	AK103	AK103	----------
Motor Oil Range Organics (MRO)	EPA 8015B/8015C8015D	EPA 8015B/8015C8015D	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Dioxins			
2,3,7,8-TeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,7,8-PeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,4,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,6,7,8-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,7,8,9-HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,4,6,7,8-HpCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
OCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
2,3,7,8-TeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
2,3,4,7,8-PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,4,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,7,8,9-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
2,3,4,6,7,8-HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,4,6,7,8-HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
1,2,3,4,7,8,9-HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
OCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total TCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total PeCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total HxCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total HeptaCDD	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total TCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total PeCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total HxCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------
Total HpCDF	EPA 8280A/8280B/8290/8290A/ 1613B	EPA 8280A/8280B/ 8290/ 8290A/1613B	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Chemical Warfare Degradates			
1,4-Dithiane	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
Benzothiazole	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
p-Chlorophenyl methylsulfide	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
p-Chlorophenyl methylsulfoxide	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
p-Chlorophenyl methylsulfone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
Chloropicrin	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
Acetophenone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
2-Chloroacetophenone	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
1,4-Oxathiane	WS-MS-0003/8270C-SIM Modified	WS-MS-0003/8270C-SIM Modified	----------
Diisopropylmethylphosphate (DIMP)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Dimethylmethylphosphonate (DMMP)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Ethyl methylphosphonic acid (EMPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Isopropyl methylphosphonic acid (IMPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Methylphosphonic acid (MPA)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Thiodiglycol (2,2'-Thiodiethanol) (TDG)	WS-LC-0004/8321A Modified	WS-LC-0004/8321A Modified	----------
Nitroaromatics			----------
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------
4-Amino-2,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------
3,5-Dinitroaniline	EPA 8330A/8330B	EPA 8330A/8330B	----------
1,3-Dinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------
2,4-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------
2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------
Glycerol trinitrate (Nitroglycerin)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330A/8330B	EPA 8330A/8330B	----------
Nitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------
2-Nitrotoluene (o-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
3-Nitrotoluene (m-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------
4-Nitrotoluene (p-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Octahydro-1,3,5,7-tetranitro1,3,5,7-tetracine (Octogen)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Picric acid	EPA 8330A/8330B	EPA 8330A/8330B	----------
Pentaerythritol Tetranitrate	EPA 8330A/8330B	EPA 8330A/8330B	----------
1,3,5-Trinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	----------
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	----------
Hexahydro-1,3-dinitroso-5-nitro-1,3,5,triazine (DNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Hexahydro-1,3,5-trinitroso-1,3,5-triazine (TNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------
1-Nitroso-3,5-dinitro-1,3,5-triazacyclohexane (MNX)	EPA 8330A/8330B	EPA 8330A/8330B	----------
Nitroguanidine	WS-LC-0010/8330A Modified	WS-LC-0010/8330A Modified	----------
Nitrosamines			
N-Nitrosodimethyl amine (NDMA)	WS-MS-0012/521 Modified	WS-MS-0012/521 Modified	----------
Perfluoro Compounds			----------
6:2 Fluorotelomer sulfonate (6:2 FTS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
8:2 Fluorotelomer sulfonate (8:2 FTS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol [N-Et-FOSE]	WS-ID-0021/8290 Modified	WS-ID-0021/8290 Modified	----------
2-(N-Methylperfluoro-1-octanesulfonamido)-ethanol [N-Me-FOSE]	WS-ID-0021/8290 Modified	WS-ID-0021/8290 Modified	----------
N-Ethyl perfluorooctane sulfonamide (EtFOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
N-Methyl perfluorooctane sulfonamide (MeFOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
N-Methyl perfluorooctanesulfon amidoacetic acide (MeFOSAA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorooctanoic acid	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorooctane Sulfonate	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorobutyric acid (PFBA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluoropentanoic acid (PFPA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Perfluorohexanoic acid (PFHxA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluoroheptanoic acid (PFHpA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorononanoic acid (PFNA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorodecanoic acid (PFDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluoroundecanoic acid (PFUDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorododecanoic acid (PFDoDA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorotridecanoic acid (PFTriA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorotetradecanoic acid (PDTeA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorobutane Sulfonate (PFBS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorohexane Sulfonate (PFHxS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluoroheptane Sulfonate (PFHpS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluorodecane Sulfonate (PFDS)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Perfluoroocatane Sulfonamide (FOSA)	WS-LC-0025/537 Modified	WS-LC-0025/537 Modified	----------
Pesticides/PCBs			
Aldrin	EPA 8081A/8081B	EPA 8081A/8081B	----------
a-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------
b-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------
d-BHC	EPA 8081A/8081B	EPA 8081A/8081B	----------
g-BHC (Lindane)	EPA 8081A/8081B	EPA 8081A/8081B	----------
a-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	----------
g-Chlordane	EPA 8081A/8081B	EPA 8081A/8081B	----------
4,4'-DDD	EPA 8081A/8081B	EPA 8081A/8081B	----------
4,4'-DDE	EPA 8081A/8081B	EPA 8081A/8081B	----------
4,4'-DDT	EPA 8081A/8081B	EPA 8081A/8081B	----------
Dieldrin	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endosulfan I	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endosulfan II	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endosulfan sulfate	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endrin	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endrin Aldehyde	EPA 8081A/8081B	EPA 8081A/8081B	----------
Endrin Ketone	EPA 8081A/8081B	EPA 8081A/8081B	----------
Heptachlor	EPA 8081A/8081B	EPA 8081A/8081B	----------
Heptachlor Epoxide	EPA 8081A/8081B	EPA 8081A/8081B	----------
Methoxychlor	EPA 8081A/8081B	EPA 8081A/8081B	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
Toxaphene	EPA 8081A/8081B	EPA 8081A/8081B	----------
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B	----------
PCB (Aroclors)			
PCB-1016	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1232	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1242	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1254	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1260	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1262	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB-1268	EPA 8082/8082A	EPA 8082/8082A	TO-4A/TO-10A
PCB (congeners) – removed references to BZ and IUPAC numbers.			
PCB 1	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 2	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 3	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 4	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 5	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 6	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 7	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 8	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 9	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 10	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 11	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 12	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 13	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 14	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 15	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 16	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 17	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 18	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 19	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 20	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 21	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 22	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 23	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 24	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 25	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 26	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 27	EPA 1668A/1668C	EPA 1668A/1668C	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 28	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 29	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 30	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 32	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 31	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 33	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 34	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 35	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 36	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 37	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 38	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 39	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 40	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 41	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 42	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 43	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 44	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 45	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 46	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 47	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 48	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 49	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 50	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 51	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 52	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 53	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 54	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 55	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 56	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 57	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 58	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 59	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 60	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 61	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 62	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 63	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 64	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 65	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 66	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 67	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 68	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 69	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 70	EPA 1668A/1668C	EPA 1668A/1668C	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 71	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 72	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 73	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 74	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 75	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 76	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 77	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 78	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 79	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 80	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 81	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 82	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 83	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 84	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 85	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 86	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 87	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 88	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 89	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 90	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 91	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 92	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 93	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 94	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 95	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 96	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 97	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 98	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 99	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 100	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 101	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 102	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 103	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 104	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 105	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 106	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 107	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 108	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 109	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 110	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 111	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 112	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 113	EPA 1668A/1668C	EPA 1668A/1668C	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 114	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 115	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 116	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 117	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 118	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 119	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 120	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 121	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 122	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 123	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 124	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 125	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 126	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 127	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 128	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 129	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 130	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 131	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 132	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 133	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 134	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 135	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 136	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 137	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 138	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 139	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 140	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 141	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 142	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 143	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 144	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 145	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 146	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 147	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 148	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 149	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 150	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 151	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 152	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 153	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 154	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 155	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 156	EPA 1668A/1668C	EPA 1668A/1668C	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 157	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 158	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 159	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 160	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 161	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 162	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 163	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 164	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 165	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 166	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 167	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 168	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 169	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 170	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 171	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 172	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 173	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 174	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 175	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 176	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 177	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 178	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 179	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 180	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 181	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 182	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 183	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 184	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 185	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 186	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 187	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 188	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 189	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 190	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 191	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 192	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 193	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 194	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 195	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 196	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 197	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 198	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 199	EPA 1668A/1668C	EPA 1668A/1668C	----------

Parameter/Analyte	Non-Potable Water	Solid/Hazardous Waste	Air
PCB 200	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 201	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 202	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 203	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 204	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 205	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 206	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 207	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 208	EPA 1668A/1668C	EPA 1668A/1668C	----------
PCB 209	EPA 1668A/1668C	EPA 1668A/1668C	----------
Metals Digestion			
Acid Digestion Total Rcoverable or Dissolved Metals	EPA 3005A	----------	----------
Acid Digestion for Total Metals	EPA 3010A	----------	----------
Acid Digestion of Sediments, Sludges and Soils	----------	EPA 3050B	EPA 3050B
Organic Preparation Methods			
Separatory Funnel Liquid-Liquid Extraction	----------	EPA 3510C	----------
Ultrasonic Extraction	----------	EPA 3550B/3550C	----------
Waste Dilution	EPA 3580A	EPA 3580A	----------
Solid-Phase Extraction	EPA 3535A	----------	----------
Volatiles Purge and Trap	EPA 5030B/5030C	EPA 5030B	----------
Volatiles Purge and Trap for Solids	----------	EPA 5035/5035A	----------
Semivolatiles in Air	----------	----------	TO-13
Chemical Warfare Degradates (in solid)	----------	WS-OP-0005	----------
Microwave Assisted Extraction	----------	EPA 3546	----------
Organic Cleanup Procedures			
Florisil Cleanup	EPA 3620B/3620C	EPA 3620B/3620C	EPA 3620B/ 3620C
Sulfur Cleanup	EPA 3660A	EPA 3660A	EPA 3660A
Sulfuric Acid Cleanup	EPA 3665A	EPA 3665A	EPA 3665A
Silica Gel Cleanup	EPA 3630C	EPA 3630C	----------

Parameter/Analyte	Potable Water
Perfluoro Compunds	
Perfluorobutane Sulfonate (PFBS)	EPA 537
Perfluoroheptanoic acid (PFHpA)	EPA 537
Perfluorohexane Sulfonate (PFHxS)	EPA 537
Perfluorononanoic acid (PFNA)	EPA 537
Perfluorooctanoic acid (PFOA)	EPA 537
Perfluoroocatane Sulfonate (PFOS)	EPA 537

Accredited Laboratory

A2LA has accredited

TESTAMERICA SACRAMENTO

West Sacramento, CA

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 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.0 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 30th day of December 2015.

Senior Director of Quality & Communications
For the Accreditation Council
Certificate Number 2928.01
Valid to January 31, 2017
Revised May 6, 2016

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

APPENDIX C
LABORATORY STANDARD OPERATING PROCEDURES
(provided on electronic copy only)

SOP QA REVIEW FORM

EMAX-300.0	Rev. 11	ION CHROMATOGRAPHY ANALYSIS
SOP No.	Revision Number	Title

SECTION	✓	COMMENTS
Scope & Application	✓	
Summary of Method	✓	
Detection Limits	✓	
Dynamic Range	✓	
Sample Holding Time & Preservation	✓	
Associated SOPs	✓	
Safety	✓	
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 necessary.

Reviewed By [signature] Date: 5/17/16

FARINA MADAMBA

STANDARD OPERATING PROCEDURES

ION CHROMATOGRAPHY ANALYSIS

SOP No.:	EMAX-300.0	Revision No.	11	Effective Date:	03-Dec-15
Prepared By:	Tu Nisamaneepong			Date:	12-03-15
Approved By:	Kenette Pimentel QA Manager			Date:	12.03.15
Approved By:	Caspar Pang Laboratory Director			Date:	12-3-15

Control Number: 300.0-11

1.0 SCOPE AND APPLICATION

1.1. This method is applicable for determination of inorganic anions in surface water, mixed domestic and industrial waste waters, ground waters and reagent waters, solids and leachates by Ion Chromatography.

1.2. List of analytes determined and established in this SOP are:

1.2.1. Part A - fluoride, chloride, bromide, nitrite-N, nitrate-N, ortho-phosphate-P, sulfate and iodide

1.2.2. Part B - bromate and chlorate.

1.2.3. Total Nitrate-Nitrite by calculation.

1.2.4. Other inorganic anions may be analyzed provided that qualitative identification and quantitative determination is established through demonstration of capability.

1.3. This SOP is an adaptation of EPA Method 300.0.

2.0 SUMMARY OF METHOD

2.1. A small amount of sample (20 µl) is injected into an ion chromatograph. The sample is passed through a guard column, anion exchange column, suppressor device and the anions are detected by a conductivity detector.

2.2. **Interferences**

2.2.1. Direct Chromatographic Coelution – occurs when a non-target analyte elutes very close to the retention time of target analytes. This interference may be solved by changing the column, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems or selective removal of the interference.

2.2.2. Concentration Dependant Coelution – occurs when the response of the neighboring peak overlaps into the retention time window of the target anion. Sample dilution or eluent dilution may resolve this interference.

2.2.3. Ionic Character Displacement – occurs when the retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening the target analytes' retention times. Sample dilution or eluent dilution may resolve this interference.

2.2.4. Method Interferences – may be caused by reagents, glassware and other substance that the sample may get in contact during the analytical process. It is imperative that reagent water production is monitored. Only reagent grade or better reagents are use. Eluate must be passed through 0.2 µm filter. Columns must be sufficiently flushed.

2.2.5. Anion Retention in the Column – anion may temporarily adhere into the column and may elute in the retention time window of fluoride and potentially interfere. Fluoride concentration above 1.5 mg/L may not be significant but the analyst must be sure that the number reported is not a false positive.

2.2.6. Carry Over Peaks – may be caused by a preceding sample with high concentration or a late eluting compound. Ozonated and chlorine dioxide matrices were observed to have unknown analytes that elutes at approximately 23 minutes. Extending the runtime to 24 minutes to allow elution of any interfering peaks may eliminate potential carry over of late eluting peaks.

3.0 DETECTION LIMITS

3.1. Detection Limits (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

3.1.1. Refer to EMAX-QA04 for generation, validation and verification of DL, LOD and LOQ.

3.1.2. Refer to Table 1 for established limits.

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 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 & PRESERVATION

5.1. Preservation

5.1.1. Water samples received in the lab are expected to be contained in either HPDE or glass bottles and cooled at ≤ 6°C without freezing.

5.1.2. Soil samples received in the lab are expected to be contained in either glass jars or Shelby tubes and cooled at ≤ 6°C without freezing.

5.1.3. Store samples and extracts to ≤ 6°C without freezing until analysis is completed.

5.2. Holding Time

5.2.1. Water Samples

Analyte	Chemical Preservative	Holding Time	
		Unpreserved	Preserved
Fluoride	None	28 days	N/A
Chloride	None	28 days	N/A
Nitrite-N	None	48 hours	N/A
Bromide	None	28 days	N/A

Analyte	Chemical Preservative	Holding Time	
		Unpreserved	Preserved
Nitrate-N	None	48 hours	N/A
Ortho-Phosphate-P	None	48 hours	N/A
Sulfate	None	28 days	N/A
Iodide	None	28 days	N/A
Nitrate/Nitrite-N	pH ≤ 2 with H_2SO_4	48 hours	28 days
Bromate	None	28 days	N/A
Chlorate	None	28 days	N/A

5.2.2. Soil Samples

5.2.2.1. Extract samples within 28 days from sampling date.

5.2.2.2. Holding time of extracts are the same as water samples.

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 for Chemicals

6.6. EMAX-QC02 Analytical Standard Preparation

6.7. EMAX-QC04 Balance Calibration

6.8. EMAX-QC06 Volumetric Labware and Micropipette Verification

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 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 samples 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 tap water. If irritations persist inform your supervisor immediately so proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS AND REAGENTS

8.1. Instrument

	Instrument D0	Instrument D7	Instrument E3
IC	Metrohm 761 Compact IC	Metrohm IC 800 Series	Methrohm 881 Compact IC Pro
Guard Column	Metrosep RP Guard		
Column	Metrosep A Supp 5 (100/4.0 mm)	Metrosep A Supp 5 (150/4.0mm)	Metrosep A Supp 7 (250x4 mm) 5µm
Detector	Detector Block		
Autosampler	788 IC Filtration Sample Processor	838 Advanced Sample Processor	858 Professional Sample Processor
Data Acquisition	IC Net 2.3 SR6		Magic Net 1.1 Ver. 2.3
Balance	Toploading, sensitive to ± 0.0001 g		

8.2. Supplies

Autosampler vials	11 ml PP sample tubes
Filters	0.022 µm Gelman IC acrodisc 4485 or equivalent
Syringes	Plastic, 5 ml
Volumetric Flasks	100, 200, 1000, 2000 ml
Containers	50 ml, 125 ml plastic snap seal
Micropipettes	1 and 5 ml; 200 µl

8.3. Chemicals and Reagents – Where available, purchase reagent-grade or better chemicals and reagents.

Reagent water	Anion-free and interference-free water
Silica Sand	Anion-free and interference-free sand
$NaHCO_3$	Bicarbonate Eluent Stock Solution 100 mM $NaHCO_3$, Reagent grade available commercially or Dissolve 8.4g $NaHCO_3$ to 1 L of reagent water, assign 1-year expiration.
Na_2CO_3/ $NaHCO_3$	Carbonate and Bicarbonate Eluent Stock Solution Instrument: D0 320/100 mM, Reagent grade available commercially or Dissolve 33.92 g of sodium carbonate (Na_2CO_3) and 8.40 g of sodium bicarbonate ($NaHCO_3$) using reagent water into 1 L solution, assign 1-year expiration. Instrument: D7 350/100 mM, Reagent grade available commercially or

	Dissolve 37.10 g of sodium carbonate (Na_2CO_3) and 8.40 g of sodium bicarbonate ($NaHCO_3$) using reagent water into 1 L solution, assign 1-year expiration Instrument: E3 360 mM, Reagent grade available commercially or Dissolve 38.156 g of sodium carbonate (Na_2CO_3) using reagent water into 1 L solution, assign 1-year expiration.
Sulfuric Acid (IC Regenerant)	100 mM Reagent Grade
Working Eluent Solution	Instrument: D0 3.2 Na_2CO_3/1.6 $NaHCO_3$mM Add 20.0 ml of 320 mM Na_2CO_3 / 100 mM combined $NaHCO_3$ into a 2 L volumetric flask and dilute to the mark with reagent water, assign 1-month expiration. Intrument : D7 3.5 Na_2CO_3/2.0 $NaHCO_3$mM Add 20.0 ml of 350 mM Na_2CO_3 / 100 mM combined $NaHCO_3$ and 20 ml of 100 mM $NaHCO_3$ solution into a 2 L volumetric flask and dilute to the mark with reagent water, assign 1-month expiration. *(Note: Degas both solutions with high purity N_2 gas for a minimum of 2 hours prior use)* Instrument: E3 3.6 mM Na_2CO_3 Add 20.0 ml of 360 mM Na_2CO_3 into a 2 L volumetric flask and dilute to the mark with reagent water, assign 1-month expiration.

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 ≤ 6°C without freezing.

9.1.3. Standard concentrations stipulated in this SOP are recommended concentration levels. In the event that concentrations are changed, perform all quality control procedures, apply related acceptance criteria and obtain Supervisor approval prior to use.

9.2. Stock Standards Solution (SSS)

9.2.1. Primary Stock Standards – are purchased commercially as certified solutions. These standards are primarily used for calibration.

Stock Standard	Source	Conc. (mg/L)
Fluoride(F^-)	ERA or equivalent	1,000

Stock Standard	Source	Conc. (mg/L)
Chloride(Cl^-)	ERA or equivalent	1,000
Nitrite-N(NO^-_2-N)	ERA or equivalent	1,000
Bromide(Br^-)	ERA or equivalent	1,000
Nitrate-N(NO^-_3-N)	ERA or equivalent	1,000
Phosphate-P(o-$PO_4^=$-P)	ERA or equivalent	1,000
Sulfate($SO_4^=$)	ERA or equivalent	1,000
Iodide(I^-)	AccuStandard or equivalent	1,000
Bromate(BrO_3^-)	ERA or equivalent	1,000
Chlorate (ClO_3^-)	ERA or equivalent	1,000

9.2.2. Secondary Stock Standards **-** are purchased commercially as certified solutions from a different source. This standard is used to prepare for ICV, and LCS.

Stock Standard	Source	Conc. (mg/L)
Fluoride(F^-)	SCP Science or equivalent	1,000
Chloride(Cl^-)	SCP Science or equivalent	1,000
Nitrite-N(NO_2^--N)	SCP Science or equivalent	1,000
Bromide(Br^-)	SCP Science or equivalent	1,000
Nitrate-N(NO_3^--N)	SCP Science or equivalent	1,000
Phosphate-P(o-$PO_4^=$-P)	SCP Science or equivalent	1,000
Sulfate($SO_4^=$)	SCP Science or equivalent	1,000
Iodide(I^-)	Inorganic Ventures or equivalent	1,000
Bromate(BrO_3^-)	SCP Science or equivalent	1,000
Chlorate (ClO_3^-)	SCP Science or equivalent	1,000

9.2.3. Preparation of Intermediate Standards

9.2.3.1. Primary Intermediate Standards. Using the primary stock standards in Section 9.2.1, prepare the following:

9.2.3.1.1. For Part A analytes (F^-, Cl^-, NO_2^- - N, Br^-, NO_3^- - N, o-$PO4^{2-}$, $SO4^{2-}$, I^-): Prepare intermediate standard at 100 mg/L by diluting 10 mL of stock standard (at 1000 mg/L) to a final volume of 100 mL with reagent water.

- Nitrate, Nitrite and Phosphate are prepared fresh on the day they are used. All other analytes are prepared weekly.

9.2.3.1.2. For Part B analytes (BrO_3^-, ClO_3^-): Prepare intermediate standards at 500 µg/L by diluting 0.05 mL of stock standard (at 1000 mg/L) to final volume of 100 mL with reagent water.

- Prepare the standards fresh on the day they are used.

9.2.3.2. Secondary Intermediate Standards. Using the secondary stock standards in Section 9.2.2, prepare the intermediate standards at the same concentrations as the primary intermediate standards shown in Section 9.2.3.1.

9.2.3.3. Store all standards at ≤ 6°C without freezing.

9.3. **Initial Calibration Standard (ICAL)**

9.3.1. Prepare a minimum of three Initial Calibration Standards and a calibration blank (S0) in 100 ml volumetric flask. Additional calibration points may be added and/or other concentrations may be used to meet project specific requirements.

9.3.2. Suggested initial calibration standards are shown in Table 2.

9.4. **Initial Calibration Verification (ICV)**

9.4.1. Using the secondary intermediate standards, prepare the ICV standard as shown in Table 2.

9.5. **Daily Continuing Calibration (DCC)**

9.5.1. Prepare DCC standard from the primary intermediate standards as shown in Table 2. Use this standard for continuing calibration check (CCC) and ending calibration check (ECC).

9.6. **Laboratory Control Standard and Matrix Spike Standard (LCS/MS)**

9.6.1. Use the secondary intermediate standards to prepare the LCS/MS Spike Standard as shown in Table 2.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Water Samples

10.1.1.1. Identify the samples for the batch (not to exceed 20 field samples) and input them in the sequence table.

10.1.1.2. Withdraw the samples from the sample control room and allow to equilibrate to room temperature.

10.1.1.3. Using a 5 ml plastic syringe, withdraw an aliquot of the sample and attach the 0.22 µm filter. Discard the first 2 ml and collect the rest in the properly labeled snap-seal container. Repeat this until sufficient amount of aliquot is collected.

10.1.1.4. Do this for all samples. Use reagent water for MB and LCS.

10.1.1.5. Take clean autosampler vials equal to the number of samples to be analyzed. Add vials for DCC, MB, LCS, MS, Duplicate, CCV and CCB.

10.1.1.6. Transfer approximately 10 ml of aliquot sample into the autosampler vials.

10.1.1.7. Spike LCS and MS designated vials with LCS/MS spike standard.

10.1.2. Soil Samples

10.1.2.1. Allow sample to equilibrate to room temperature.

10.1.2.2. Mix sample thoroughly discarding artifacts, e.g. vegetation, rocks, etc.

10.1.2.3. Tare a 125 ml polyethylene bottle to nearest 0.1 g.

10.1.2.4. Weigh 10 g of homogenized soil sample to the nearest 0.1 g. For MB and LCS, use silica sand.

10.1.2.5. 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.

10.1.2.6. Remove bottles from the shaker and centrifuge the slurry for 15-30 minutes or allow the particles to settle.

10.1.2.7. Treat the liquid phase like an aqueous sample and follow Sections 10.1.1.3 to 10.1.1.7.

10.2. **Instrument Parameters**

	Inst. D0	Inst. D7	Inst. E3
Instrument	Metrohm		
Detector	Conductivity		
Column	Metrosep A Supp 5 (100/4.0 mm)	Metrosep A Supp 5 (150/4.0 mm)	Metrosep A Supp 7 250x4.0 mm
Sample Flow Rate	0.70 ml/min	0.70 ml/min	0.8 ml/min
Pump Pressure (Mpa)	5.0-6.0	6.5-7.5	9.5-10.5
Sample Loop	20 µl		
Run Time	w/o I= 14.0 min; w/ I= 19min.	w/o I= 16.0 min; w/ I= 25min.	30 min
Scale Setting (µS)	1000	100	2 µS/cm
Oven Temp	45°C for E3 only		

10.3. **Calibration**

10.3.1. Instrument Set Up

10.3.1.1. Set up the Metrohm IC to the proper operation parameter established in Section 10.2.

10.3.1.2. Prime the pump to eliminate air bubbles in the system.

10.3.1.3. Step the suppressor thrice and equilibrate the column for 30 minutes or until a stable baseline is obtained.

10.3.2. Initial Calibration (ICAL)

10.4.3.1 Prepare initial calibration standards as suggested in Table 2. Additional calibration standards may be analyzed when deemed necessary.

10.3.2.1. Analyze the ICAL standards. Establish the individual analyte calibration curve by plotting the peak area against the known concentration.

10.3.2.2. Calculate the slope and intercept of the linear curve.

10.3.2.3. Refer to Appendix 1 for acceptance criteria.

10.3.2.3.1. If the correlation coefficient (r) is out of acceptance criteria, check the results again and identify which calibration point is off.

10.3.2.3.2. If the highest ICAL point appears to be bias high, exclude the highest point.

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Analyze ICV from second source after the initial calibration to verify the validity of the initial calibration.

10.3.3.2. Refer to Appendix 1 for acceptance criteria.

10.3.4. Retention Rime Window (RTW)

10.3.4.1. After initial calibration, establish a tentative retention time window based from the calibration points.

10.3.4.2. Tabulate the retention time for each analyte and calculate their standard deviations. Retention time window is determined by ± 3 X standard deviation.

10.3.4.3. Determine actual retention time window from the continuing calibrations within the course of a day. RTW is determined by 3X standard deviation of retention time for each analyte.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze initial calibration check standard and initial calibration blank (ICC/ICB) at the beginning of analytical sequence.

10.3.5.2. Analyze continuing calibration check standard (CCC) after every 10 samples or whenever the anion eluent is changed or as per project requirements.

10.3.5.3. Analyze a calibration check blank (CCB) after every calibration check standard.

10.3.5.4. Analyze ending calibration check standard (ECC) at the end of each analytical sequence of a 24-hour shift.

10.3.5.5. Refer to Appendix 1 for acceptance criteria and corrective action.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. ICV – initial calibration check

10.4.1.2. ICB – initial calibration blank (reagent water)

10.4.1.3. MB – method blank

10.4.1.4. LCS – lab control sample

10.4.1.5. LCSD – lab control sample duplicate

10.4.1.6. Samples – maximum of 10 field samples

10.4.1.7. MS – matrix spike sample

10.4.1.8. MSD or Dup – matrix spike sample duplicate or duplicate

10.4.1.9. CCV – continuing calibration check

10.4.1.10. CCB – continuing calibration blank

10.4.1.11. Samples – maximum of 10 field samples

10.4.1.12. ECC – ending calibration check

10.4.1.13. ECB – ending calibration blank

10.4.2. Sample Result Evaluation

10.4.2.1. Check that the ICV, MB and LCS are within control limits. Refer to Appendix 1 for acceptance criteria and corrective action.

- Check the MB for absence or presence of contamination.
- Check the LCS for method performance.
- Check Dup to establish the precision of the method.
- Check the MS for absence or presence of matrix interference.
- Check MSD or Dup to establish the precision of the method.

10.4.2.2. Qualitatively identify detected analytes using the actual retention time window. Check that the retention time for all positive results fall within the actual RTW as described in Section 10.3.4.

10.4.2.3. 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.
- If manual integration is necessary, retain the original chromatogram and the corrected chromatogram must be initialed and dated by the analyst and the supervisor.
- If a peak appears to be cryptic / anomalous, consult the supervisor.

10.4.2.4. Check that results exceeding calibration range are appropriately diluted and re-analyzed at a concentration within the calibration range.

10.4.2.5. Rule-out any suspicion of carry-over. Re-analyze any sample with trace amount of analyte(s) seen in a previous sample that exceeds the calibration range.

10.4.2.6. Properly fill up the analytical run log.

10.5. **Calculations**

10.5.1. Calculate for Concentration

10.5.1.1. Water Samples

$$Cw = (mA + b) * DF \qquad \text{Eq.-10.5.1.1.}$$

where:

Cw – Concentration of analyte in sample, in mg/L

m – Slope of the linear calibration curve

A – Peak area of the analyte in sample

b – Intercept of the linear calibration curve

DF – Dilution Factor = V_f / V_i

where V_f – final volume of diluted sample, ml

V_i – initial volume of diluted sample, ml

10.5.1.2. Soil Samples

$$Cs = \frac{(mA + b) * PF * DF}{\%S}$$

where:

Cs – Concentration of analyte in sample, in mg/Kg

m – Slope of the linear calibration curve

A – Peak area of the analyte in sample

b – Intercept of the linear calibration curve

PF – Preparation Factor = *Ve / W*

Where *Ve* – final extract volume, ml

W – sample amount, g

DF – Dilution Factor = V_f / V_i

where V_f – final volume of diluted extract, ml

V_i – initial volume of extract, ml

$\%S$ – Percent Solids = 1- (%Moisture/100)

10.5.2. Calculate Total Nitrate/Nitrite – N

$$Total\ Nitrate/Nitrite\text{-}N = Nitrate\text{-}N + Nitrite\text{-}N \qquad \text{Eq.-10.5.2}$$

where: *Nitrate-N and Nitrite-N* are derived separately from Eq. 10.5.1.1.

10.5.3. Calculate for Total Nitrate/Nitrite

$$Total\ Nitrate/Nitrite = Nitrate\text{-}N \times (MW_{NO3} / MW_N) + Nitrite\text{-}N \times (MW_{NO2} / MW_N) \qquad \text{Eq.-10.5.3}$$

where:

MW_{NO3} – molecular weight of Nitrate = 62

MW_{NO2} – molecular weight of Nitrite = 46

MW_N – molecular weight of Nitrogen = 14

10.5.4. Calculate for Percent Recovery

$$\%Recovery = \frac{C_f - C}{C_s} \times 100 \qquad \text{Eq.-10.5.2}$$

where:

C_f – Concentration Found

C – Concentration of Sample (use 0 for LCS)

C_s – Concentration of spike

10.5.5. Calculate for Precision

$$RPD = \frac{|C_1 - C_2|}{\frac{C_1 + C_2}{2}} \times 100 \qquad \text{Eq.-10.5.3}$$

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.

10.6.2. Make a copy of the sample preparation log (if any), refer to Form 300.0FS page 1.

10.6.3. Print a copy of the sample preparation log (if any), refer to sample preparation log page 2.

10.6.4. Print a copy of the raw data and the QC report.

10.6.5. Highlight the data to be reported.

10.6.6. Collate the reportable raw data separating the QC results from the sample results.

10.6.7. 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 and sample results using ANIONSF1.exe.

10.7.2. Generate the Sample Duplicate, LCS and MS summaries using ANIONSF3.exe.

10.7.3. Generate the case narrative using CN00.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.

10.8.1.1. Case Narrative

10.8.1.2. Sample Results

10.8.1.3. LCS/LCSD Summary

10.8.1.4. MS/MSD Summary

10.8.1.5. Sample Duplicate Result Summary (if any)

10.8.1.6. ICAL Summary

10.8.1.7. ICV Summary

10.8.1.8. DCC Summary

10.8.1.9. Analytical Run Log

10.8.1.10. Sample Preparation Log (if any)

10.8.1.11. Retention Time Summary

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.

- ✓ Check that identifications are done properly.
- ✓ Check that sample results are integrated properly and results over calibration range are diluted and re-analyzed within calibration range.
- ✓ Check that presence of saturated peak(s) are diluted, and quantitated properly.
- ✓ Check that suspected carry-overs are confirmed.
- ✓ Review the attached logs that they are properly filled.
- ✓ Check the generated reports against the raw data. 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. Perform daily routine check and record it in the instrument maintenance log. Refer to Form 300.0FM for daily routine maintenance check points. This practice ensures optimum operating condition of the equipment thus reducing the possibility of frequent instrument malfunction.

10.9.2. The table below is a list of preventive maintenance activities that are essential in performing this SOP.

Maintenance Activity	Description	Frequency
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
IC Pump Maintenance	Replace pump head seal and filter. Perform wear-in procedure and leak test	Every six months or as necessary
Column Maintenance	Replace column or flush column as necessary.	As necessary

	Perform pressure test.	
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 tubing. Lubricate moving parts.	Once a year or as necessary
Inspection	Perform general inspection of the complete system	Once a year

10.9.3. Maintain an inventory of instrument parts and supplies for routinely maintenance.

11.0 QUALITY CONTROL

11.1. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Pipettes must have a valid calibration prior to use. Refer to EMAX-QC06 for details.

11.3. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.4. Acceptance criteria are summarized in Appendix 1.

11.5. Assessing Analytical QC. Every analytical batch is expected to consist of the following to comply with the requirement:

- Daily Continuing Calibration (DCC, CCV and ECV)
- Method Blank (MB)
- Lab Control Sample (LCS)

11.6. Assessing Analyte Recovery Data Quality on a given matrix is demonstrated for each group of sample with similar matrix and is expected to consist of the following to comply with the requirement:

- Matrix Spike (MS)
- MS spike level must be high enough to be detected above the original sample and not less than 4 times the LOD.
- If the concentration of fortification is less than 25% of the background concentrations measured in the unfortified sample, the matrix recovery is not calculated.
- If recovery for MS falls outside the recovery range and the LCS is within control, the recovery problem for MS is judged to be matrix related, and not system related.

11.7. Perform MDL study every 6 months. Quarterly verification of LOD and LOQ must be performed. Refer to EMAX-QA04 for details.

11.8. Perform quarterly verification of calibration standards and instrument performance with the analysis of a Quality Control Sample (QCS). Since the QCS is equivalent to the LCS, this verification is done on every batch.

11.9. 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 action for each Quality Control Procedure is summarized in Appendix 1.

12.2. **Sample Preparation QC**

12.2.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.2.2. When method blank is non-compliant, investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.

- If the analyte found in the method 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.

12.3. **Sample Analysis QC**

12.3.1. When Continuing Calibration Verification (CCV) 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.

12.3.3. If the reagent water shows contamination in the instrument or in the method blank consider changing the filters of the reagent water source.

12.3.4. If the QC'd blank soil shows contamination in the method blank, consider baking the soil at 400°C overnight then repeat QC prior to its use. If problem persists, discontinue the use of the lot and use a new QC'd lot.

12.4. **Method QC**

12.4.1. When LOQ verification is non-compliant, consider instrument maintenance and/or repeating the MDL study.

12.4.2. When retention time significantly shifts, check for any bubbles or leaks.

12.5. 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 reagents that may go to the wastewater drains.

13.2. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples maybe 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. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

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. **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 MS is analyzed. For every analytical batch at least one MB and one LCS is analyzed.

15.1.3.2. **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.

15.1.4. 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%.

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. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.8. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.9. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.10. Method Blank (MB) – 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.

15.1.11. 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.

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. 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.

15.1.20. 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, MB or LCS.

15.1.21. Raw Data - Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, 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.

15.2. **Application of EMAX QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Ion Chromatography analysis unless. 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 or 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. EPA Method 300.0, Methods for Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)

16.2. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Tables**

17.1.1. Table 1 Established DL, LOD and LOQ Limits

17.1.2. Table 2 Calibration Standard Preparation

17.2. **Figures**

17.2.1. Figure 1 Typical Peak Evaluation

17.2.2. Figure 2A Typical Chromatogram

17.2.3. Figure 2B Typical Bromate and Chlorate Chromatograms

17.2.4. Figure 3 Typical Sample Result Summary

17.2.5. Figure 4 Typical LCS/LCSD Summary

17.2.6. Figure 5 Typical MS/MSD Summary

17.2.7. Figure 6 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. 300.0FS Sample Preparation Log

17.4.2. 300.0FA Analytical Run Log

17.4.3. 300.0FM Instrument Maintenance Log

Table 1: ESTABLISHED DL, LOD AND LOQ LIMITS

PARAMETER	WATER (mg/L)			SOIL (mg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
Bromide	0.125	0.25	0.5	1.25	2.5	5
Chloride	0.05	0.1	0.2	0.5	1	2
Fluoride	0.025	0.05	0.1	0.25	0.5	1
Nitrate	0.025	0.05	0.1	0.25	0.5	1
Nitrite	0.025	0.05	0.1	0.25	0.5	1
Phosphate	0.125	0.25	0.5	1.25	2.5	5
Sulfate	0.125	0.25	0.5	1.25	2.5	5
Iodide	0.125	0.25	0.5	1.25	2.5	5

PARAMETER	WATER (µg/L)		
	DL	LOD	LOQ
Bromate	2.5	5	10
Chlorate	2.5	5	10

Table 2: CALIBRATION STANDARD PREPARATION

Analyte	Inter-mediate Standard Conc.	S0	S1		S2		S3		S4		S5		ICV / DCC		LCS / MS	
		Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.	Int. Std. Aliquot (mL)	Conc.
Fluoride (F^-)	100mg/L	0	0.1	0.1mg/L	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	2.5	2.5 mg/L	2	2 mg/L
Chloride (Cl^-)	100mg/L	0	0.2	0.2mg/L	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	2	2 mg/L	2	2 mg/L
Nitrite-N (NO^-_2-N)	100mg/L	0	0.1	0.1mg/L	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	2.5	2.5 mg/L	2	2 mg/L
Bromide (Br^-)	100mg/L	0	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	20	20mg/L	5	5mg/L	2	2 mg/L
Nitrate-N (NO^-_3-N)	100mg/L	0	0.1	0.1mg/L	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	2.5	2.5 mg/L	1	1mg/L
Phosphate-P (o-$PO_4^=$-P)	100mg/L	0	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	20	20mg/L	5	5mg/L	2	2 mg/L
Sulfate ($SO_4^=$)	100mg/L	0	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	20	20mg/L	5	5mg/L	5	5mg/L
Iodide (I^-)	100mg/L	0	0.5	0.5mg/L	1	1mg/L	5	5mg/L	10	10mg/L	20	20mg/L	5	5mg/L	2	2 mg/L
Bromate (BrO_3^-)	500µg/L	0	2	10µg/L	10	50µg/L	20	100µg/L	50	250µg/L	100	500µg/L	20	100µg/L	20	100µg/L
Chlorate (ClO_3^-)	500 µg/L	0	2	10µg/L	10	50µg/L	20	100µg/L	50	250µg/L	100	500µg/L	20	100µg/L	20	100µg/L

* Use Primary Intermediate Standard for ICAL and DCC Standards. Use Secondary Intermediate Standard for ICV and LCS/MS Standards. All standards are prepared to a ***final volume of 100 mL*** using reagent water.

Figure 1: TYPICAL PEAK EVALUATION

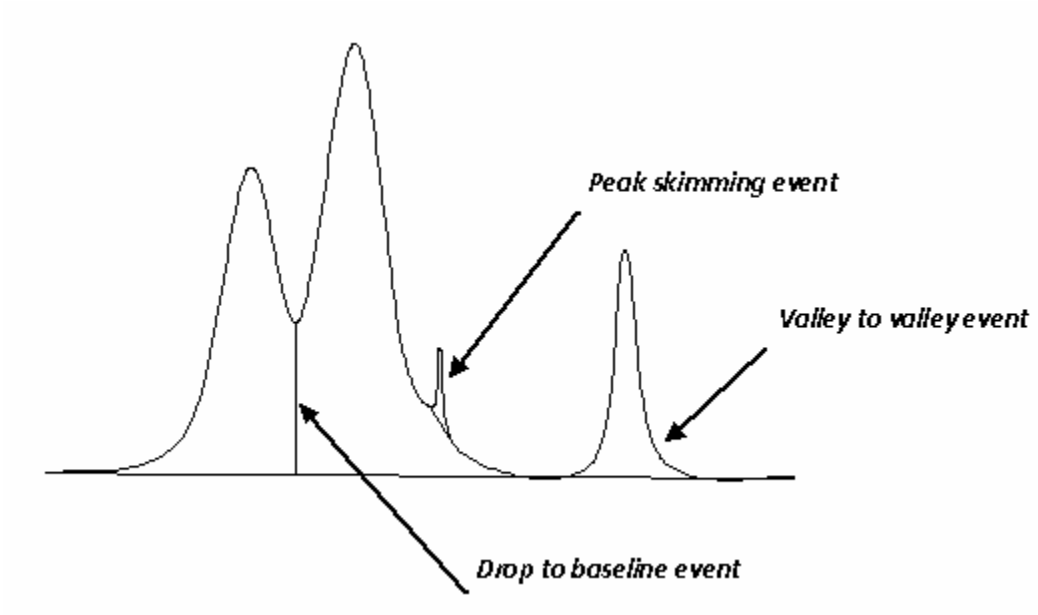


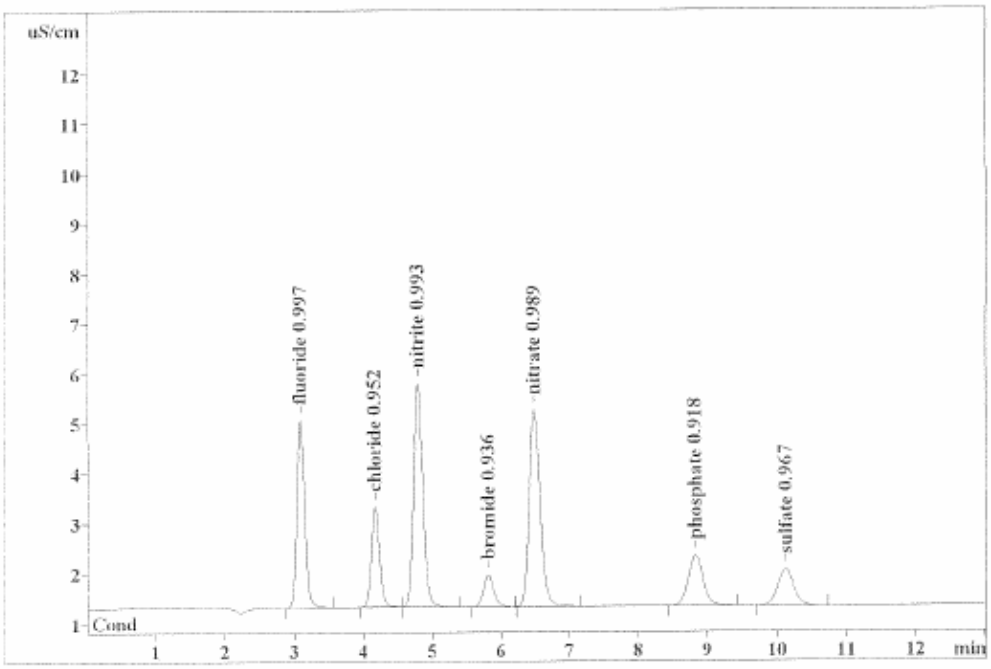
Figure 2A: **TYPICAL CHROMATOGRAM**

Report date: 7/31/2013 2:02:22 PM
Printed by: Jadelyn Chun

Ident: AG43-12 ICV
Analysis from: 7/31/2013 12:50:28 PM
File: x7311250.chw Last save: 7/31/2013 1:03:25 PM

Method: ICD0-G31.mtw Last save: 7/31/2013 12:45:19 PM
Run operator: Jadelyn Chun
Analysis number: 90999

SAMPLE: 1.00PPM
:
Vial number: 12
Volume: 1.0 µL
Dilution: 1.00
Amount: 1.0000



Quantitation method: Custom

No	Retention min	Height uS/cm	Area uS/cm*sec	Conc. mg/L	Name
1	3.09	3.74	29.508 ✓	0.997 ✓	fluoride
2	4.17	2.00	16.288 ✓	0.952 ✓	chloride
3	4.79	4.46	41.290 ✓	0.993 ✓	nitrite
4	5.81	0.63	6.532 ✓	0.936 ✓	bromide
5	6.48	3.93	42.966 ✓	0.989 ✓	nitrate
6	8.82	1.01	15.091 ✓	0.918 ✓	phosphate
7	10.12	0.72	11.482 ✓	0.967 ✓	sulfate
7	13.00	16.50	163.158	6.752	

This report has been created by IC Net
METROHM LTD

AA
07/31/13

Figure 2B: TYPICAL BROMATE & CHLORATE CHROMATOGRAMS

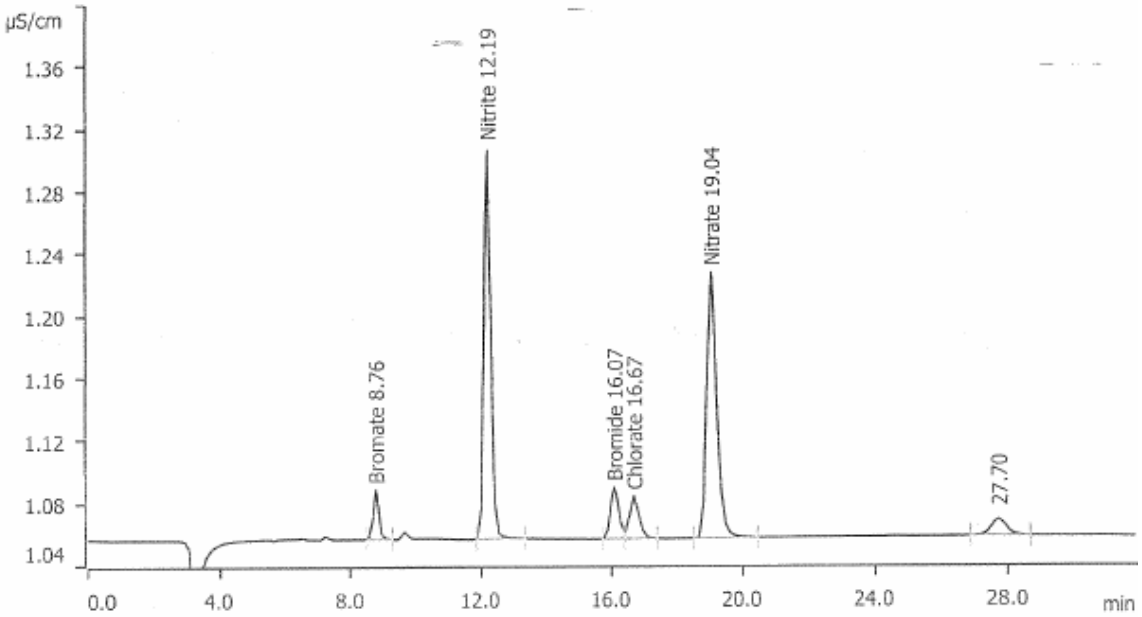
Mag - Net

2013-07-09 12:54:14 PM

Sample data

Sequence E3G08-10
Ident ICV
Determination start 2013-07-08 19:38:14 UTC-7
Method . . . . . E3G0813 METHOD
Sample type . . . . . Check standard 1
Dilution Factor 1
Operator . . . . . KYAW

Anions



Peak number	Retention time min	Area (µS/cm) x min	Height µS/cm	Component name	Concentration ppb
1	8.763	0.005941	0.032	Bromate	100.749
2	12.185	0.056397	0.249	Nitrite	98.459
3	16.070	0.009834	0.033	Bromide	97.921
4	16.670	0.009361	0.027	Chlorate	97.254
5	19.042	0.060018	0.171	Nitrate	100.328
6	27.698	0.005386	0.011		invalid

AS
07/11/13

Metrohm

Page 1 of 1

Figure 3: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD 300.0
NITRATE-N

Client : XYZ, INC.
Project : CLEAN WATER
Batch No. : 13H046

Matrix : WATER
Instrument ID : D0

SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	DLF	MOIST	LOQ (mg/L)	DL (mg/L)	LOD (mg/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Coll DATETIME	Rec DATE
MBLK1W	ICH005WB	ND	1	NA	0.100	0.0250	0.0500	08/07/1310:22	NA	AH05-03	AH05-01	ICH005W	NA	NA
LCS1W	ICH005WL	1.00	1	NA	0.100	0.0250	0.0500	08/07/1310:37	NA	AH05-04	AH05-01	ICH005W	NA	NA
LCD1W	ICH005WC	1.00	1	NA	0.100	0.0250	0.0500	08/07/1310:52	NA	AH05-05	AH05-01	ICH005W	NA	NA
SB(8-6-13)	H046-04	ND	1	NA	0.100	0.0250	0.0500	08/07/1315:01	NA	AH05-08	AH05-06	ICH005W	08/06/1310:35	08/07/13
RB(8-6-13)	H046-05	ND	1	NA	0.100	0.0250	0.0500	08/07/1315:16	NA	AH05-09	AH05-06	ICH005W	08/06/1310:45	08/07/13
MWBG-5(8-6-13)	H046-02I	13.1	10	NA	1.00	0.250	0.500	08/07/1315:31	NA	AH05-10	AH05-06	ICH005W	08/06/1310:05	08/07/13
DUP-1(8-6-13)	H046-03I	13.2	10	NA	1.00	0.250	0.500	08/07/1315:47	NA	AH05-11	AH05-06	ICH005W	08/06/1310:10	08/07/13
MWBG-4(8-6-13)	H046-01J	9.56	20	NA	2.00	0.500	1.00	08/07/1319:49	NA	AH05-27	AH05-21	ICH005W	08/06/1312:10	08/07/13
MWBG-4(8-6-13)DUP	H046-01JD	9.61	20	NA	2.00	0.500	1.00	08/07/1320:04	NA	AH05-28	AH05-21	ICH005W	08/06/1312:10	08/07/13
MWBG-4(8-6-13)MS	H046-01JM	31.2	20	NA	2.00	0.500	1.00	08/07/1320:20	NA	AH05-29	AH05-21	ICH005W	08/06/1312:10	08/07/13
MWBG-4(8-6-13)MSD	H046-01JS	31.2	20	NA	2.00	0.500	1.00	08/07/1320:35	NA	AH05-30	AH05-21	ICH005W	08/06/1312:10	08/07/13

Figure 4: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 13H046
METHOD: METHOD 300.0

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	ICH005WB	ICH005WL	ICH005WC		
LAB FILE ID:	AH05-03	AH05-04	AH05-05		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	NA
DATE ANALYZED:	08/07/1310:22	08/07/1310:37	08/07/1310:52	DATE RECEIVED:	NA
PREP. BATCH:	ICH005W	ICH005W	ICH005W		
CALIB. REF:	AH05-01	AH05-01	AH05-01		

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 (%)
Nitrate-N	ND	1	1	100	1	1	100	0	90-110	15

Figure 5: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 13H046
METHOD: METHOD 300.0

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	20	20	20		
SAMPLE ID:	MWBG-4(8-6-13)				
LAB SAMP ID:	H046-01J	H046-01JM	H046-01JS		
LAB FILE ID:	AH05-27	AH05-29	AH05-30		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	08/06/13 12:10
DATE ANALYZED:	08/07/1319:49	08/07/1320:20	08/07/1320:35	DATE RECEIVED:	08/07/13
PREP. BATCH:	ICH005W	ICH005W	ICH005W		
CALIB. REF:	AH05-21	AH05-21	AH05-21		

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 (%)
Nitrate-N	9.56	20	31.2	108	20	31.2	108	0	80-120	15

Figure 6: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER

SDG : 13H046

METHOD 300.0
NITRATE BY IC

A total of five (5) water samples were received on 08/07/13 for Anions by IC analysis, Method 300.0 in accordance with Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100).

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 ICH005WL/C were all within QC limits.

Matrix QC Sample
Matrix QC sample was analyzed at the frequency prescribed by the project.
Percent recoveries for H046-01M/H046-01S are 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 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Initial Calibration Min. 3 pts + CB	Every 6 months or when CCV fails to meet the acceptance criteria.	Correlation coefficient ≥ 0.995 for linear regression	Correct the problem then repeat initial calibration		
Initial Calibration Verification (ICV)	After every initial calibration and every 3 months.	All analytes within ± 10% of expected value	Correct the problem then repeat initial calibration		
Continuing Calibration Verification (CCV)	At the beginning of analytical batch and every 10 samples thereafter and at the end of the sequence.	All analytes within ± 10% of expected value.	Re-analyze DCC. If re-analysis is still outside acceptance limits, discontinue the analysis. Resolve the cause of failure and re-calibrate prior to resuming analysis.		
Method Blank (MB)	One per preparation batch	No analytes detected > ½ LOQ. Blank result must not affect sample results.	Re-prep and re-analyze MB and all samples processed with MB.		
Lab Control Sample (LCS)	One LCS per preparation batch	All analytes within ± 10% of expected value.	Re-prep and re-analyze the LCS and all associated samples		
Matrix Spike (MS)	One per analytical batch unless specified by the project.	All analytes within project QC Limits or within ± 20% of expected value.	If LCS passed, no action.		
MDL Study	Every 6 months	Refer to EMAX-QA04.	None.		
Comments: For flagging criteria refer to PSR.			Reviewed By		
			Date		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
ANIONS BY ION CHROMATOGRAPHY

MATRIX: WATER

SOP: EMAX-300.0 Rev. 10
Conc Unit: mg/L
Analyzed date: 1/7/2015
Analyzed by: J. Chun

PARAMETER	AA05-04	AA05-05	AA05-31	AA05-32	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	ICA005WL	ICA005WC	ICA007WL	ICA007WC							
Bromide	1.92	1.91	1.90	1.90	2	1.93	96	0.007	0.4	90 - 110	PASSED
Orthophosphate	1.94	1.96	1.91	1.93	2	1.93	97	0.022	1.1	90 - 110	PASSED
Sulfate	4.73	4.73	4.77	4.73	5	4.74	95	0.020	0.4	90 - 110	PASSED
Fluoride	2.07	2.06	2.06	2.07	2	2.06	103	0.005	0.3	90 - 110	PASSED
Chloride	1.83	1.83	1.82	1.83	2	1.83	91	0.008	0.4	90 - 110	PASSED
Nitrite	2.01	2.00	2.00	2.01	2	2.00	100	0.005	0.3	90 - 110	PASSED
Nitrate	1.01	1.01	1.01	1.01	1	1.01	101	0.003	0.3	90 - 110	PASSED

SOP: EMAX-300.0 Rev. 10
Conc Unit: µg/L
Analyzed date: 1/6/2015
Analyzed by: K. Linn

PARAMETER	E3A05-04	E3A05-05	E3A05-06	E3A05-07	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	BCA001WL	BCA001WC	BCA001WX	BCA001WY							
Bromate	104	104	104	104	100	103.10	103	0.087	0.1	90 - 110	PASSED
Chlorate	104	105	104	105	100	104.49	104	0.793	0.8	90 - 110	PASSED

MATRIX: SOIL

SOP: EMAX-300.0 Rev. 10
Conc Unit: mg/Kg
Analyzed date: 1/8/15 & 1/22/15
Analyzed by: J. Chun

PARAMETER	AA05-59	AA05-60	AA27-18	AA27-19	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	ICA002SX	ICA002SY	ICA003SL	ICA003SC							
Bromide	18.8	18.8	18.8	18.9	20	19.1	95	0.017	0.1	90 - 110	PASSED
Orthophosphate	19.0	19.3	19.2	19.2	20	19.2	96	0.109	0.6	90 - 110	PASSED
Sulfate	46.6	46.9	49.8	50.9	50	48.5	97	2.138	4.4	90 - 110	PASSED
Fluoride	20.6	20.6	20.8	20.7	20	20.7	103	0.097	0.5	90 - 110	PASSED
Chloride	18.1	18.1	19.0	19.5	20	18.7	93	0.677	3.6	90 - 110	PASSED
Nitrite	19.9	20.0	20.1	20.1	20	20.0	100	0.087	0.4	90 - 110	PASSED
Nitrate	10.1	10.1	10.0	10.0	10	10.1	101	0.074	0.7	90 - 110	PASSED

SOP: EMAX-300.0 Rev. 10
Conc Unit: µg/Kg
Sample Amount(g): 10
Extract Volume (mL): 100
Analyzed date: 1/6/2015
Analyzed by: K. Linn

PARAMETER	E3A05-15	E3A05-16	E3A05-17	E3A05-18	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	BCA001SL	BCA001SC	BCA001SX	BCA001SY							
Bromate	1049	1042	1047	1046	1000	1037	104	2.6	0.3	90 - 110	PASSED
Chlorate	1015	1035	1055	1061	1000	1041	104	20.9	2.0	90 - 110	PASSED

300.0FS: SAMPLE PREPARATION LOG (Page 1)

EMAX LABORATORIES, INC

SAMPLE PREPARATION LOG *for* ION CHROMATOGRAPHY

Page 1

Note: For samples and relevant QCs/Standards extracted, refer to attached extraction sequence.

Comments:

Book #: EIC-005

Preparation Batch:

Matrix:

Balance ID:

Pipette ID:

Filter:

SOP	Rev. #
☐ EMAX-300.0	10
☐ EMAX-4110B	5
☐ EMAX-9056	6

Standards	ID	Amount Added (ml)
Fluoride		
NO_2-N		
NO_3-N		
Chloride		
Br^-		
PO_4P		
SO_4^{2-}		

Reagent	Lot # / ID
Sand	
Reagent Water	

Prepared By:

Standard Added By:

Checked By:

300.0FS: SAMPLE PREPARATION LOG (Page 2)

EMAX LABORATORIES, INC.

FileID: IC.xls

PREPARATION LOG FOR ANIONS (SOIL)

PrepBatchID	LabSampleID	Aliquot	Unit	DateTime	FinalVol (ml)	ExpAmt	ExpVf (ml)	PrepFctr	Comments

PrepFctr=(ExpAmt/Aliquot)*(Vf/ExpVf)

Comments:

Prepared By:

Checked By:

Date

300.0FA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG *for* ION CHROMATOGRAPHY

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #:	AD7-022
Instrument No.:	D7
Pipette ID's:	039380124
	SW3-02-02-02
	SW8A-02-13
Analytical Sequence:	
Method File:	
Analytical Batch:	

SOP #	Rev. #
☐ EMAX-300.0	11
☐ EMAX-4110B	5
☐ EMAX-9056	3
☐ EMAX-	
STANDARDS ID	
ICAL	
ICV	
CCV	
LCS	
MS	

Filters Lot #:	Snap Seal Containers Lot #:
0.45 µm:	4 oz:
0.2 µm:	1.5 oz:

Column: Metrosep A Supp 5-150

Flow Rate: 0.70 ml/min

IC ELUENT PREPARATION Expiration Date:

Std ID	Aliquot	Final Vol*
	20 mL	2 L
	20 mL	

IC REGENERANT PREPARATION Expiration Date:

Std ID	Aliquot	Final Vol*
	100 mL	1 L

ELECTRONIC DATA ARCHIVAL	
Location	Date
☐ IC-METROHM	
☐ External Hard Drive	

Analyzed By:

Date:

* Reagent Water ID:

300.0FM: INSTRUMENT MAINTENANCE LOG

Page 1

EMAX LABORATORIES, INC

INSTRUMENT MAINTENANCE LOG

Instrument No: D7 Month: Year: 201_ Book #: MD7-003

Date							
Background Noise (<0.01)							
Check Eluent bottle level							
Check DI bottle level							
Check H_2SO_4 bottle level							
Check autosampler rinse beakers							
Pump Setting at 0.70 ml/min							
Pump Pressure							
uS Reading							
Autosampler Filter							
Printer Paper							
Computer Clock							
Analyst Initial							
Troubleshooting							
Problem(s) Encountered							
Return To Specs (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

SOP REVIEW FORM

EMAX-300M	Rev. 3	ION CHROMATOGRAPHY ANALYSIS FOR ORGANIC ACIDS
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 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 L.A. [illegible] LUCITA ARZADON. Date: 06/18/15

STANDARD OPERATING PROCEDURES

ION CHROMATOGRAPHY ANALYSIS FOR ORGANIC ACIDS

SOP No.:	EMAX-300M	Revision No.	3	Effective Date:	03-Oct-12
Prepared By:	Lucita Arzadon			Date:	10/02-12
Approved By:	Kenette Pimentel QA Manager			Date:	10.02.12
Approved By:	Caspar Pang Laboratory Director			Date:	10-02-12

Control Number: 300M-03-

1.0 SCOPE AND APPLICATION

1.1. This method is applicable for the determination of the following organic acids: lactate, acetate, propionate, butyrate and pyruvate in drinking water, surface water, mixed domestic and industrial waste waters, ground waters, reagent waters, solids, and leachate by Ion Chromatography.

1.2. This SOP is an adaptation of EPA Method 300.0.

2.0 SUMMARY OF METHOD

2.1. A small amount of sample (25 µl) is injected into an ion chromatograph. The sample is passed through a guard column, ion exchange column, suppressor device and the organic acids are detected by a conductivity detector.

2.2. **Interferences**

2.2.1. Direct Chromatographic Coelution – occurs when a non-target analyte elutes very close to the retention time of target analytes. This interference may be solved by changing the column, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems or selective removal of the interference.

2.2.2. Concentration Dependant Coelution – occurs when the response of the neighboring peak overlaps into the retention time window of the target anion. Sample dilution or eluent dilution may resolve this interference.

2.2.3. Ionic Character Displacement – occurs when the retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening the target analytes' retention times. Sample dilution or eluent dilution may resolve this interference.

2.2.4. Method Interferences – may be caused by reagents, glassware and other substance(s) that the sample may get in contact during the analytical process. It is imperative that reagent water production is monitored. Only reagent grade or better reagents are used. Eluate must be passed through 0.2 µm filter. Columns must be sufficiently flushed.

2.2.5. Pretreatment Cartridges – can be an effective eliminator of matrix interference. However, artifacts are known to leach from certain cartridges. If samples are passed through pretreatment cartridge, all instrument calibration standards must be treated the same.

2.2.6. Anion Retention in the Column – Anion may temporarily adhere into the column and may elute in the retention time window of organic acids and potentially interfere.

2.2.7. Carry Over Peaks – may be caused by a preceding sample with high concentration or a late eluting compound. Ozonated and chlorine dioxide matrices were observed to have unknown analytes that elute at approximately 23 minutes. Extending the runtime to 40 minutes to allow elution of any interfering peaks may eliminate potential carry over of late eluting peaks.

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 of DL, LOD and LOQ.

3.1.2. Refer to Table 1 for established limits.

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 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 & PRESERVATION

5.1. Sample Collection

5.1.1. Water samples received in the lab are expected to be contained in either HPDE or glass bottles and cooled at ≤ 6°C without freezing.

5.1.2. Soil samples received in the lab are expected to be contained in either glass jars or Shelby tubes and cooled at ≤ 6°C without freezing.

5.2. Holding Time

5.2.1. Water Samples

Analyte	Chemical Preservative	Holding Time
Lactate	None	28 days
Acetate	None	28 days
Propionate	None	28 days
Butyrate	None	28 days
Pyruvate	None	28 days

5.2.2. Soil Samples

5.2.2.1. Extract samples within 28 days from sampling date.

5.2.2.2. Holding time of extracts are the same as water samples.

5.3. **Preservation**

5.3.1. Store samples and extracts to ≤ 6°C without freezing until analysis is completed.

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 for 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 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 samples 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 tap water. If irritations persist inform your supervisor immediately so proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS AND REAGENTS

8.1. **Instrument**

IC	Dionex ICS-1000 Ion Chromatography
Guard Column	AG16
Column	AS11-HC
Detector	Conductivity Cell
Autosampler	Dionex, AS 50
Data Acquisition	Chromeleon 6.70 Build 1820
Balance	Toploading, sensitive to ± 0.0001 g

8.2. **Supplies**

Autosampler vials	2 ml with filter caps
Filters	0.2 µm & 0.45 µm PTFE Membrane or equivalent
Matrix Pretreatment Cartridge	Barium form – OnGuard Ba cartridge 046072 or equivalent Silver form – OnGuard Ag cartridge 039637 or equivalent Hydrogen form – OnGuard H cartridge 039596 or equivalent
Volumetric Flasks	100 ml, 250 ml, 1000 ml, 2000 ml
Sample Containers	Plastic snap seal containers 50 ml & 125 ml
Microliter pipettes	100 & 1000 µl; 5 & 10 ml

8.3. **Chemicals and Reagents**

Reagent water	De-ionized water (ASTM Type II) or equivalent
Eluent stock 60mM	50% w/w NaOH: Add 9.6 g of NaOH to a 2 L volumetric flask and dilute to the mark with reagent water or commercially reagent grade solution may be used. This solution must be purged with Nitrogen for at least 10 minutes prior to use.
NaOH	50% NaOH W/V grade, available commercially
1.8 mM Eluent Solution	Add 60 ml of 60mM NaOH solution to a 2 L volumetric flask and dilute to the mark with reagent water
Gas	Nitrogen, 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 in refrigerator at ≤ 6°C without freezing. Follow specific manufacturing instructions, if provided (e.g., Lactate – Refrigerate 2-10°C).

9.2. **Stock Standards Solution (SSS)**

9.2.1. Primary Stock Standards - are purchased as neat standards from which a 1000 mg/L is prepared. These standards are primarily used for calibration.

Primary Stock Standards					Final Conc. (mg/L)
Analyte	**Source**	**Conc. (mg/L)**	**Aliquot (g)**	**Final Volume (ml)**	
Lactate	J. T. Baker # K38665	60%	1.048	500	1,000
Acetate	E. M. Science # 40073052	Assay	0.695	500	1,000
Propionate	J. T. Baker # N20596	Assay	0.657	500	1,000
Butyrate	J. T. Baker # L12663	Assay	0.632	500	1,000
Pyruvate	Spectrum #PF0237	Assay	0.632	500	1,000

9.2.2. Secondary Stock Standards - are purchased commercially as certified solutions from a different source. This standard is used to prepare for ICV and QCS.

Secondary Stock Standard					Final Conc. (mg/L)
Analyte	**Source**	**Conc. (mg/L)**	**Aliquot (ml)**	**Final Volume (ml)**	
Lactate	E. M. Science # 36267648	60%	1.048	500	1,000
Acetate	J. T. Baker # K26168	Assay	0.695	500	1,000
Propionate	Spectrum # S0192	Assay	0.657	500	1,000
Butyrate	AVOCADO # H6373B	Assay	0.632	500	1,000
Pyruvate	MALLINCKRODT # 2694V43H07	Assay	0.632	500	1,000

9.2.3. Preparation of Intermediate Standard

9.2.3.1. Intermediate standard, 100 mg/L. Dilute 10 ml of stock solution to 100 ml with reagent water.

9.3. **Initial Calibration Standards (ICAL)**

9.3.1. Using a micropipette, prepare a minimum of 3-point Initial Calibration Standards (ideally 5-point) from the stock standard and a method blank using a 100 ml volumetric flask. For a 5-point calibration, the following concentrations are recommended. Additional calibration points may be added and/or the concentrations may be varied as long as the acceptance criteria are met.

ICAL POINTS	Final Concentration (mg/L)				
	Lactate	Acetate	Propionate	Butyrate	Pyruvate
S_0	0	0	0	0	0
S_1	0.1	0.1	0.1	0.1	0.1
S_2	0.5	0.5	0.5	0.5	0.5
S_3	1.0	1.0	1.0	1.0	1.0
S_4	2.0	2.0	2.0	2.0	2.0
S_5	3.0	3.0	3.0	3.0	3.0

9.4. **Initial Calibration Verification Standard (ICV)**

9.4.1. Use the secondary stock solution to prepare ICV at a spike level of S_4.

9.5. **Continuing Calibration Verification Standard (CCV)**

9.5.1. Use the primary stock standard for CCV at a spike level equivalent to S_4. This solution is used for ICV, CCV and Ending Calibration Verification (ECV).

9.6. **LCS/MS Spike Standard**

9.6.1. Prepare a working standard to fortify LCS from the neat standard, spiked at S_4 level and diluted to a final volume of 100 ml with reagent water.

9.6.2. MS is spiked at S_3 level.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Water Samples

10.1.1.1. Identify the samples for the preparation batch and record them in the sample preparation log.

10.1.1.2. Withdraw the samples from the sample control room and allow the samples to equilibrate to room temperature.

10.1.1.3. Rinse filter with reagent water.

10.1.1.4. Using a 5 ml plastic syringe, withdraw 10 ml of sample and attach the 0.45 µm filter and, if needed, post filter with pre-treatment cartridge (On Guard Ba, On Guard Ag & On Guard H). Discard the first 2 ml and collect the rest on a properly labeled sample container, volume to equal to 2 ml.

10.1.1.5. Repeat steps 10.1.3 and 10.1.4 for all samples. Do the same for the MB and LCS using reagent water.

10.1.1.6. Take clean sample vials equal to the number of samples to be analyzed not to exceed 20 field samples. If number of samples are ≤ 10 add vials for CCVs, MB, LCS and MS. If samples > 10 add three more vials for CCC, CCB, and MS.

10.1.1.7. Spike S_3 level of MS standard to designated vials.

10.1.1.8. Seal the vials and shake the QC samples to attain homogeneity on the mixture.

10.1.2. Soil Sample

10.1.2.1. Allow the samples to equilibrate to room temperature.

10.1.2.2. Mix the sample thoroughly.

10.1.2.3. Tare a 125 ml snap seal bottle to the nearest 0.1 g.

10.1.2.4. Weigh 10 grams of the mixed soil to the nearest 0.1 g.

10.1.2.5. Using a 100 ml grade 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.

10.1.2.6. Remove the bottle from the shaker and centrifuge the slurry for 15 to 30 minutes or allow the particles to settle.

10.1.2.7. Treat the liquid phase like an aqueous sample.

10.2. **Instrument Parameters**

Instrument	ICS-1000
Detector	Conductivity
GP Flow Rate	1.5 ml/min
GP Pressure	0-3000-psi
ASRS-4mm	100 mA
Loop Size	25 µl
AS50 Autosampler Tray temperature	20°C
Eluent Pressure	1.80mM NaOH, 4 psi
Nitrogen generator pressure	100-psi

10.3. **Calibration**

10.3.1. Instrument Set Up

10.3.2. Set up the ICS-1000 with the proper operation parameter established in Section 10.3.

10.3.2.1. Prime the pump to eliminate air bubbles in the system and equilibrate the column for 30 minutes or until a stable baseline is obtained.

10.3.3. Initial Calibration (ICAL)

10.4.3.1 Prepare initial calibration solution as suggested in section 9.3.1. Additional points may be analyzed when deemed necessary.

10.3.3.1. Analyze the ICAL as described in section 10.4.

10.3.3.2. Refer to section 10.5 for calculations.

10.3.3.3. Check Appendix 1 for acceptance criteria.

10.3.3.4. If the correlation coefficient (r) is out of acceptance criteria, check the results again and identify which one is off.

10.4.3.4.1 If the highest ICAL point appears to be bias high, exclude the highest point

10.3.4. Retention Rime Window (RTW)

10.3.4.1. After initial calibration, establish a tentative retention time window based from the calibration points.

10.3.4.2. Tabulate the retention time for each analyte and calculate their standard deviations. Retention time window is determined by ± 3x standard deviation.

10.3.4.3. Actual retention time window is established based on the continuing calibrations within the course of a 24-hour shift as described in 10.4.4.2.

10.3.5. Initial Calibration Verification (ICV)

10.3.5.1. Analyze ICV from second source after the initial calibration to verify the validity of the initial calibration and every 3 months thereafter.

10.3.6. Continuing Calibration Verification (CCV)

10.3.6.1. To verify the calibration curve, perform CCV by analyzing the initial calibration check standard.

10.3.6.2. Analyze ICV standard and initial calibration blank (ICB) at the beginning of the 24-hour shift.

10.3.6.3. Analyze continuing calibration check standard (CCC) and continuing calibration blank after every 10 samples or whenever the anion eluent is changed.

10.3.6.4. Analyze ending calibration check standard (ECC) and ending calibration blank at the end of each analytical sequence of a 24-hour shift.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. ICV – initial calibration check

10.4.1.2. ICB – initial calibration blank (reagent water)

10.4.1.3. MB – method blank

10.4.1.4. LCS – lab control sample

10.4.1.5. MS –matrix spike

10.4.1.6. Samples – maximum of 10 field samples

10.4.1.7. CCV – continuing calibration verification

10.4.1.8. CCB – continuing calibration blank

10.4.1.9. MS – another lab fortified matrix sample

10.4.1.10. Samples – maximum of 10 field samples

10.4.1.11. ECC – ending calibration check

10.4.1.12. ECB – ending calibration blank

10.4.2. All sample runs, including the MB, LCS, Sample Duplicate (Dup), and MS are bracketed with calibration checks.

10.4.3. If any analyte concentration exceeds the initial calibration range, perform appropriate dilution to bring the concentration within the range and re-analyze the dilution. All re-analyses due to dilution are bracketed with continuing calibrations.

10.4.4. Sample Result Evaluation

10.4.4.1. Check each of the instrument performance checks that each one meets the acceptance criteria set forth in Appendix 1.

10.4.4.2. Check the retention time for all positive results that they fall within the established RTW.

10.4.4.3. 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.
- If manual integration is necessary refer to EMAX-DM01 (Manual Integration Section). Retain the original chromatogram, and the corrected chromatogram must be initialed and dated by the analyst and supervisor.
- If peak appears to be cryptic/anomalous, consult the supervisor.

10.4.4.4. Check if any of the sample results exceeds the calibration range. If such results exist, dilute the sample or extract and re-analyze.

10.4.4.5. 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.

10.4.4.6. Check the MB for absence or presence of contamination.

10.4.4.7. Check the LCS for method performance.

10.4.4.8. Check the MS for absence or presence of matrix interference.

10.4.4.9. Properly fill up the analytical run log.

10.5. **Calculations**

10.5.1. Calculation 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:

$$Cw = (C)(DF) \qquad \text{Eq.-10.5.1}$$

where:

C_w	–	Concentration in original sample, in mg/L
C	–	Computer generated concentration in diluted digestate in mg/L
DF	–	Dilution factor = V_f/V_i
V_i	–	Initial volume of the sample, in ml
V_f	–	Final volume of diluted sample, in ml

10.5.2. Calculation for Solid Samples

$$Cs = C \text{ x } \frac{Ve}{Ws} \text{ x } DF \text{ x } \frac{100}{100 - \%M} \qquad \text{Eq.-10.5.2}$$

where:

C_s	–	Concentration of the sample based on dry weight in mg/Kg
C	–	Computer generated concentration in diluted digestate in mg/L
V_e	–	Volume of extract, mg/L
W_s	–	Weight of "wet sample" in g
DF	–	Dilution factor of extract; DF = V_f/V_i
V_i	–	Initial volume of the sample extract, in ml
V_f	–	Final volume of diluted sample extract, in ml
$\%M$	–	Percent Moisture of "wet sample"

If the dilution factor and ratio of extract volume/sample weight are entered in the sequence run table file, the concentration is represented in wet weight.

10.5.3. Calculate for Percent Recovery

$$\%Recovery = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.5.3}$$

where:

C_f – Concentration found

C – Concentration of the sample (use 0 for LCS)

C_s – Concentration of spike

10.5.4. Calculate for Precision

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C1 + C2}{2}\right)} x100 \qquad \text{Eq.-10.5.4}$$

where:

RPD – Relative Percent Difference

C_1 – Measured concentration of the first sample aliquot

C_2 – Measured concentration of the second sample aliquot

10.6. **Data Reduction**

10.6.1. Make a copy of the analytical run log.

10.6.2. Make a copy of the sample preparation log (if any).

10.6.3. Print a copy of the raw data and the QC report.

10.6.4. Highlight the data to be reported.

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 WDBX[1].exe.

10.7.2. Generate the sample results using MSRBX.exe

10.7.3. Generate the LCS and MS summary using QCIC.exe.

10.7.4. Generate the sample duplicate result using CQ1N.exe.

10.7.5. Generate the case narrative using CN2.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

[1] X – version number

10.8.1.2. Sample Results

10.8.1.3. LCS/LCSD Summary

10.8.1.4. MS/MSD Summary

10.8.1.5. Sample Duplicate Result Summary (if any)

10.8.1.6. ICAL Summary

10.8.1.7. ICV Summary

10.8.1.8. DCC Summary

10.8.1.9. Analytical Run Log

10.8.1.10. Sample Preparation Log (if any)

10.8.1.11. Retention Time Summary

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.

10.8.3. Check Qualitative Identification

- ✓ Check that the actual RTW for the analytical batch was done properly.
- ✓ Check that positively identified peaks are integrated properly.
- ✓ Initial and date manual integration(s) to confirm that integration was done properly.
- ✓ Check that suspicion of carry-over (if any) was ruled out.

10.8.4. Check Quantitation

- ✓ Since a program generates the sample result forms, check the calculation of one sample result for correctness.
- ✓ Check that dilution factors are properly factored in the calculation.
- ✓ Check that correct sample amount and/or extract amount is properly factored in.

10.8.5. Review the attached logs that they are properly filled.

10.8.6. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.7. 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.

10.8.8. 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. Refer to Form 300MFM for daily routine maintenance check points. This practice ensures optimum operating condition of the equipment thus reducing the possibility of frequent instrument malfunction.

10.9.2. The table below is a list of preventive maintenance activities that are essential to consider in performing this SOP.

Maintenance Activity	Description	Frequency
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
IC 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 or flush column 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 tubing. Lubricate moving parts.	Once a year or as necessary
Inspection	Perform general inspection of the complete system	Once a year

10.9.3. Maintain an inventory of instrument part and supplies for routinely maintenance.

11.0 QUALITY CONTROL

11.1. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06 for details.

11.3. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.4. Acceptance criteria is summarized in Appendix 1.

11.5. Assessing Analytical QC. Every analytical batch is expected to consist of the following to comply with the requirement:

- Continuing Calibration Verification (CCV)
- Method Blank (MB)
- Lab Control Sample (LCS)

11.6. Assessing Analyte Recovery Data Quality on a given matrix is demonstrated for each group of sample with similar matrix and is expected to consist of the following to comply with the requirement:

- Matrix Spike (MS)
- MS spike level must be high enough to be detected above the original sample and not less than 4 times the LOD.
- If the concentration of fortification is less than 25% of the background concentrations measured in the unfortified sample, the matrix recovery is not calculated.
- If recovery for MS falls outside the recovery range and the LCS is within control, the recovery problem for MS is judged to be matrix related, and not system related.

11.7. A valid DL and LOD must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.8. 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 action for each Quality Control Procedure is summarized in Appendix 1.

12.2. **Sample Preparation QC**

12.2.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.2.2. When method 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 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-extract and re-analyze the method blank with the associated samples.

12.2.4. If solvents and reagents that did not undergo quality control check were accidentally used, consider the following to correct the problem.

- Check that there was no data integrity impact. Otherwise, repeat the analysis of all associated samples with new QC samples using QC'd solvent and/or reagent.
- If method blank is clean, use that data to document the reagent QC.

12.3. **Sample Analysis QC**

12.3.1. When CCV is non-compliant, correct the problem and repeat CCV. If problem persists, consult with the Supervisor for further advice prior to performing a new ICAL.

12.3.2. When there is carry-over, consider running a rinse.

12.3.3. If the reagent water shows contamination in the instrument or method blank consider changing the filters of the reagent water source.

12.3.4. If the QC'd blank soil shows contamination in the method blank, consider baking the soil at 400°C overnight then re-QC prior to its use. If problem persists, discontinue the use of the lot and use a new QC'd lot.

12.4. **Method QC**

12.4.1. When LOD verification is non-compliant, consider instrument maintenance and/or repeating the LOD study.

12.4.2. When retention time significantly shifts, check the gas flow or check for leaks.

12.5. A Non-Compliance Report (NCR) is required when the following circumstances occur:

- Sample container is broken and no other sample can be analyzed.
- Samples were wrongly preserved.
- 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. Quantity of chemicals purchased is based on expected usage during its shelf life to minimize disposal of unused material.

13.2. Actual reagent preparation volumes, reflects anticipated usage and reagent stability.

13.3. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.4. Prepare all standards in fume hoods.

14.0 WASTE MANAGEMENT

14.1. No samples maybe 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 the 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.

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.

15.1.1.2. **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.

15.1.2. 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%.

15.1.3. 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%.

15.1.4. Limit of Quantitation (LOQ) – is at the lowest concentration that produces a quantitative result within a specified limits of precision bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

15.1.5. Material Safety Data Sheet (MSDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.6. Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

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. 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.

15.1.11. 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.

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. 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 analysis for organic acids by ion chromatography. 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 Method 300.0, Methods for the Determination of Inorganic Substance in Environmental Samples

16.2. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Tables**

17.1.1. Table 1 Established Limits

17.2. **Figures**

17.2.1. Figure 1 Typical Peak Evaluation Technique

17.2.2. Figure 2 Typical Chromatogram

17.2.3. Figure 3 Typical Sample Result Summary

17.2.4. Figure 4 Typical LCS/LCSD Summary

17.2.5. Figure 5 Typical MS/MSD Summary

17.2.6. Figure 6 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. 300MFS Sample Preparation Log

17.4.2. 300MFA Analytical Run Log

17.4.3. 300MFM Instrument Maintenance Log

Table 1: ESTABLISHED LIMITS

PARAMETER	WATER (mg/L)		
	DL	LOD	LOQ
Lactate	0.025	0.05	0.10
Acetate	0.025	0.05	0.10
Propianate	0.025	0.05	0.10
Butyrate	0.025	0.05	0.10
Pyruvate	0.04	0.05	0.10

Figure 1: TYPICAL PEAK EVALUATION TECHNIQUE

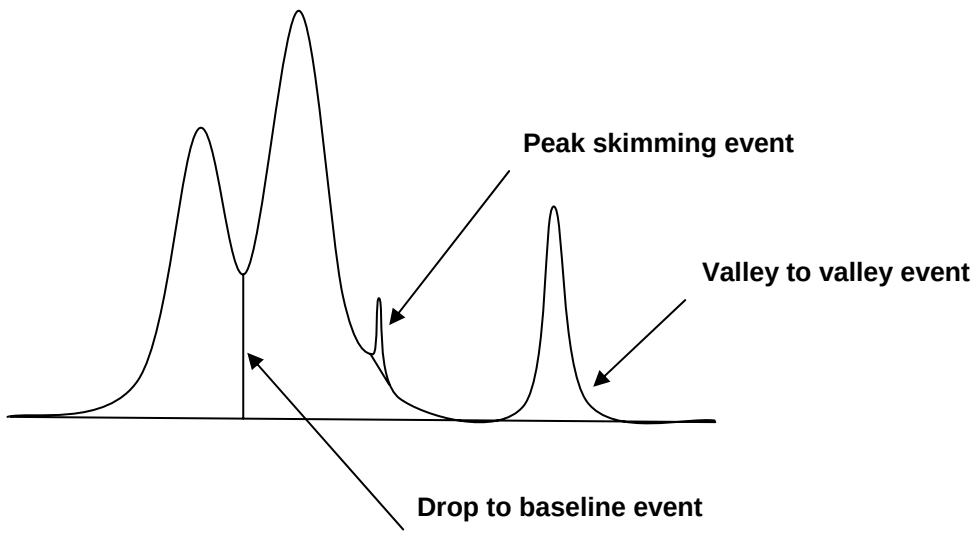


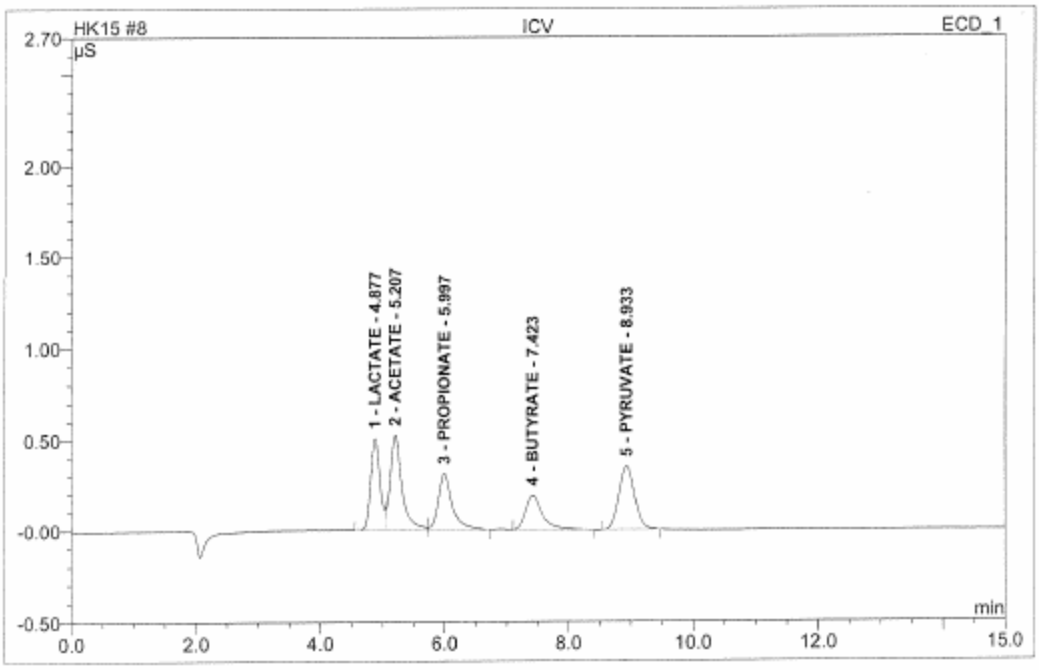
Figure 2: **TYPICAL CHROMATOGRAM**

Operator:cmadmin Timebase:ICS1000 Sequence:HK15

Page 1-1
11/16/2011 12:57 PM

HK15 008 ICV

Sample Name:	**ICV**	*Injection Volume:*	**25.0**
Vial Number:	**38**	*Channel:*	**ECD_1**
Sample Type:	**unknown**	*Wavelength:*	**n.a.**
Control Program:	**ORG**	*Bandwidth:*	**n.a.**
Quantif. Method:	**OAD6K15**	*Dilution Factor:*	**1**
Recording Time:	**11/15/2011 20:01**	*Sample Weight:*	**1.0000**
Run Time (min):	**15.00**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height µS	Area µS*min	Rel.Area %	Amount	Type
1	4.88	LACTATE	0.504694	0.088011	19.68	1.990	BM
2	5.21	ACETATE	0.520464	0.117058	26.18	2.054	M
3	6.00	PROPIONATE	0.309202	0.081457	18.22	2.074	MB
4	7.42	BUTYRATE	0.190046	0.060090	13.44	1.937	BMB
5	8.93	PYRUVATE	0.347528	0.100546	22.49	1.954	BMB
Total:			1.872	0.447	100.00	10.009	

Figure 3: TYPICAL SAMPLE RESULT SUMMARY

METHOD 300.0(M)
ACETIC ACID

Client : XYZ, INC.
Project : CLEAN WATER PROJECT
Batch No. : 11F010

Matrix : WATER
Instrument ID : D6

SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	DLF	MOIST	RL (mg/L)	MDL (mg/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK1W	OAF004WB	ND	1	NA	0.500	0.0500	06/07/1113:41	NA	HF07003	HF07001	OAF004W	NA	NA
LCS1W	OAF004WL	10.1	1	NA	0.500	0.0500	06/07/1114:02	NA	HF07004	HF07001	OAF004W	NA	NA
LCD1W	OAF004WC	10.2	1	NA	0.500	0.0500	06/07/1114:22	NA	HF07005	HF07001	OAF004W	NA	NA
25GW104-0611	F010-01	730	100	NA	50.0	5.00	06/07/1115:24	NA	HF07007	HF07001	OAF004W	06/01/1111:15	06/02/11
25GW109-0611	F010-02	790	100	NA	50.0	5.00	06/07/1116:46	NA	HF07009	HF07001	OAF004W	06/01/1111:30	06/02/11
25GW103-0611	F010-03	431	50	NA	25.0	2.50	06/07/1119:11	NA	HF07013	HF07010	OAF004W	06/01/1111:35	06/02/11
25GW106-0611	F010-04	1860	200	NA	100	10.0	06/07/1121:14	NA	HF07016	HF07010	OAF004W	06/01/1115:00	06/02/11
25GW107-0611	F010-05	2500	400	NA	200	20.0	06/07/1123:57	NA	HF07020	HF07010	OAF004W	06/01/1115:11	06/02/11
25GW105-0611	F010-07	1980	200	NA	100	10.0	06/08/1103:23	NA	HF07026	HF07021	OAF004W	06/01/1116:56	06/02/11
25GW105-0611MS	F010-07M	2900	200	NA	100	10.0	06/08/1104:04	NA	HF07027	HF07021	OAF004W	06/01/1116:56	06/02/11
25GW105-0611MSD	F010-07S	2940	200	NA	100	10.0	06/08/1104:44	NA	HF07028	HF07021	OAF004W	06/01/1116:56	06/02/11
MBLK2W	OAF002WB	ND	1	NA	0.500	0.0500	06/06/1117:20	NA	HF06003	HF06001	OAF002W	NA	NA
LCS2W	OAF002WL	10.3	1	NA	0.500	0.0500	06/06/1117:41	NA	HF06004	HF06001	OAF002W	NA	NA
LCD2W	OAF002WC	10.3	1	NA	0.500	0.0500	06/06/1118:24	NA	HF06005	HF06001	OAF002W	NA	NA
25GW108-0611	F010-08	ND	1	NA	0.500	0.0500	06/07/1100:56	NA	HF06011	HF06001	OAF002W	06/01/1116:50	06/02/11
25GW110-0611	F010-09	ND	1	NA	0.500	0.0500	06/07/1106:02	NA	HF06017	HF06012	OAF002W	06/01/1116:38	06/02/11

Figure 4: TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 11F010
METHOD: METHOD 300.0(M)

MATRIX: WATER
% MOISTURE: NA

DILUTION FACTOR:	1	1	1
SAMPLE ID:	MBLK2W		
LAB SAMP ID:	OAF002WB	OAF002WL	OAF002WC
LAB FILE ID:	HF06003	HF06004	HF06005
DATE EXTRACTED:	NA	NA	NA
DATE ANALYZED:	06/06/1117:20	06/06/1117:41	06/06/1118:24
PREP. BATCH:	OAF002W	OAF002W	OAF002W
CALIB. REF:	HF06001	HF06001	HF06001

DATE COLLECTED: NA
DATE RECEIVED: NA

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 (%)
Acetic Acid	ND	10.0	10.3	103	10.0	10.3	103	0	90-110	20

Figure 5: TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 11F010
METHOD: METHOD 300.0(M)

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	200	200	200		
SAMPLE ID:	25GW105-0611				
LAB SAMP ID:	F010-07	F010-07M	F010-07S		
LAB FILE ID:	HF07026	HF07027	HF07028		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	06/01/11 16:56
DATE ANALYZED:	06/08/1103:23	06/08/1104:04	06/08/1104:44	DATE RECEIVED:	06/02/11
PREP. BATCH:	OAF004W	OAF004W	OAF004W		
CALIB. REF:	HF07021	HF07021	HF07021		

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 (%)
Acetic Acid	1980	1000	2900	92	1000	2940	96	1	80-120	20

Figure 6: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER PROJECT

SDG : 11F010

METHOD 300.0(M)
ORGANIC ACIDS BY IC

A total of eight (8) water samples were received on 06/02/11 for Organic Acids (Acetic, Butyric, Lactic, Propionic and Pyruvic) by IC analysis, Method 300.0(M) in accordance with Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100).

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 blanks were analyzed at the frequency required by the project. For this SDG, two (2) method blanks were analyzed with the samples. All results were compliant to project requirement. Refer to QC result summary forms for details.

Lab Control Sample
Two (2) sets of LCS/LCD were analyzed with the samples in this SDG.
Percent recoveries for OAF002WL/C were all within QC limits.
Percent recoveries for OAF004WL/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 one result qualified with [*] in F010-07 M/S summary form of Lactic Acid, most likely due to matrix interference. Check QC summary form 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 PROCEDURE

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd 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 ≥ 0.995 for linear regression	Correct the problem then repeat initial calibration		
Quality Control Sample (QCS) or ICV	After every initial calibration	All analytes within ± 10% of expected value	Correct the problem then repeat initial calibration		
Instrument Performance Check (IPC) or CCV	Bracket every 10 field samples with IPC.	All analytes within ± 10% of expected value	Repeat calibration and re-analyze all samples since last successful calibration		
Laboratory Reagent Blank (LRB) or MB	One per preparation batch	No analytes detected ≥ LOD	Re-prep and re-analyze LRB and all samples processed with LRB		
Laboratory Fortified Blank or LCS	One LFB per preparation batch	%R = 90 – 110%	Re-prep and re-analyze the LCS and all associated samples		
Laboratory Fortified Matrix (LFM) or MS	One LFM per 10 field samples per matrix	%R = 80 –120%	If LFB passed, no action		
Method Detection Limit	Once every 6 months	None	None		
Comments: For flagging criteria refer to PSR.			Reviewed By:		
			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
ORGANIC ACIDS BY IC
METHOD EPA 300M

SOP: EMAX-300M
Conc Unit: µg/L
Sample Amount(ml): 25

Analysis date: 3/22/12 - 03/23/12
Analyzed by: C. Dam

PARAMETER	HC22013	HC22014	HC23003	HC23004	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	OAC006WL	OAC006WC	OAC008WL	OAC008WC							
Lactate	1.93	1.96	1.89	1.89	2	1.919	96	0.03	2	70 - 130	PASSED
Acetate	1.98	2.04	2.00	1.98	2	1.998	100	0.03	1	70 - 130	PASSED
Propionate	2.01	2.09	2.18	2.11	2	2.098	105	0.07	3	70 - 130	PASSED
Butyrate	1.90	1.84	2.14	1.98	2	1.966	98	0.13	7	70 - 130	PASSED
Pyruvate	1.99	2.08	1.91	1.96	2	1.986	99	0.07	4	70 - 130	PASSED

300MFS: **SAMPLE PREPARATION LOG**

EMAX LABORATORIES, INC

Page 1

SAMPLE PREPARATION LOG FOR ORGANIC ACID/PERCHLORATE

SOP ☐ EMAX-314.0 Rev. _ ☐ EMAX-300.0M Rev. _ ☐ EMAX- Book # E0P-003

Matrix: Start Date: Time: End Date: Time:

PREPARATION BATCH # _____

Sample Prep ID	Lab Sample ID	Sample Amount (g)	Dilution Volume (ml)	Extract Volume (ml)	Notes
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					

Standards	ID	Amount Added (ml)
Acetate		
Butyrate		
Lactate		
Propionate		
Pyruvate		
Perchlorate		

Reagent	Lot # / ID
H_2SO_4	
Nanopure	
Sand	

Legend:

Color	Texture	Clarity	Artifacts
Bu = Blue Bl = Black	Cs = Coarse	Cr = Clear	Rt = Roots
Bn = Brown Gn = Green	Md = Medium	Cy = Cloudy	Sl = Shale
Og = Orange Rd = Red	Fn = Fine	Td = turbid	Ve= Vegetation
Yw = Yellow			

Balance ID: _____

Micropipette ID: _____

Comments: _____

Prepared By: _____ Standard Added By: _____

300MFA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG *for* ION CHROMATOGRAPHY

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Eluent : 60 mM NaOH: (60 ml) - 2L reagent water

Reagent Water:

Book #:	AD6-012
Instrument No.:	D6
Pipette ID's:	SW8A-02-19
	SW8A-02-20
	SW8A-02-21
Balance ID #:	40706360
Analytical Sequence:	
Method File:	
Analytical Batch:	

SOP #	Rev. #
☐ EMAX-300.0M	
☐ EMAX-7199	
☐ EMAX-218.6	
☐ EMAX-	

STANDARDS ID	
ICAL	
ICV	
CCV	
LCS	
MS	

ELECTRONIC DATA ARCHIVAL	
Location	Date
☐ CHROMALEON	

Analyzed By:

Date:

300MFM: INSTRUMENT MAINTENANCE LOG

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: D6 Month: Year: 201_ Book #: MD6-003

Date							
Background Noise (<0.01)							
Check Eluent bottle level							
Check DI bottle level							
Check H_2SO_4 bottle level							
Check autosampler rinse beakers							
Pump Setting at 0.7ml/min							
Pump Pressure (4.5-6.5mpa)							
µS Reading							
Autosampler Filter							
Printer Paper							
Computer Clock							
Analyst Initial							
Troubleshooting							
Problem(s) Encountered							
Return To Specs (Analyst initial)							

Code	Problems Encountered	Code	Corrective Action

EMAX
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

SOP REVIEW FORM

EMAX-2320B	Rev. 3	ALKALINITY
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 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 Mary Jane Mendoza [signature] Date: 6/22/15

STANDARD OPERATING PROCEDURE

ALKALINITY

SOP No.:	EMAX-2320B	Revision No.	3	Effective Date:	17-Oct-11
Prepared By:	Mary Jane Mendoza			Date:	10.13.11
Approved By:	Kenette Pimentel QA Manager			Date:	10.13.11
Approved By:	Caspar Pang Laboratory Director			Date:	10-13-11

Control Number: 2320B-03-

1.0 SCOPE AND APPLICATION

1.1. This method is used to measure alkalinity of aqueous samples by titration. This is applicable to drinking, surface, and saline waters, domestic and industrial wastes. It is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.

1.2. Alkalinity of soil may also be determined by leaching the matrix with reagent water and analyzing the leachate for alkalinity.

1.3. This SOP is an adaptation of Method 2320B, Standard Methods for the Examination of Water and Wastewater, 20th Edition.

2.0 SUMMARY OF METHOD

2.1. An unaltered amount of sample is titrated with standard acid titrant to an end point of pH=4.5.

2.2. Solid matrices are leached with reagent water prior to titration.

2.3. **Interferences**

2.3.1. Substances, such as salts of weak organic or inorganic acids present in large amounts, may cause interference in the electrometric measurements.

2.3.2. Oil and grease coating the pH electrode may cause sluggish response. For samples having high concentration of mineral acids, such as mine wastes and associated receiving waters, ASTM Method D-1067 may be use.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL) and Limit of Detection (LOD)**

3.1.1. DL and LOD are not applicable for this method.

3.2. **Limit of Quantitation (LOQ)**

3.2.1. Refer to EMAX-QA04 for generation, validation and verification of LOQ.

3.2.2. Established LOQ for this method is 5 mg/L.

4.0 DYNAMIC RANGE

4.1. The maximum amount of titrant used in a pre-determined amount of sample shall not exceed 50 ml. All samples analyzed above these range shall be considered estimate (over range) and shall require a smaller amount of sample or higher titrant concentration to properly quantitate.

5.0 SAMPLE HOLDING TIME & PRESERVATION

5.1. **Preservation**

5.1.1. Collected water samples are maintained at ≤ 6°C with no chemical preservation.

5.1.2. Samples should not be filtered, diluted, concentrated or altered in any way.

5.1.3. Store all samples ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. Analyze aqueous samples as soon as possible but not to exceed 14 days from sampling date.

5.2.2. Analyze all leachates within 24 hours from extraction.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM01 Data Flow and Review

6.2. EMAX-QA04 Method Detection Limit

6.3. EMAX-QA05 Training

6.4. EMAX-QA08 Corrective Action

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

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 hazard. 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. All sample and standard handling shall be performed 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 AND REAGENTS

8.1. **Instruments and Supplies**

8.1.1. pH meter: Hach One Laboratory pH/ISE Meter or equivalent

8.1.2. Autotitrator: PC Titrate

8.1.3. Combination electrode

8.1.4. Burette: 50, 25 and 10 ml

8.1.5. Beaker: 50 ml

8.1.6. Volumetric flasks: 1 L and 2 L

8.1.7. Analytical balance (capable of weighing 0.01 gm)

8.2. **Chemicals and Reagents**

8.2.1. Reagent water: Type II

9.0 STANDARDS

9.1. Purchase all standards as certified solutions as listed below (or equivalent):

STANDARD	SOURCE	CONCENTRATION
pH 4.0	VWR	NA
pH 7.0	VWR	NA
pH 10.0	VWR	NA
pH 8.0	VWR	NA
Alkalinity Std.	VWR/ERA	Certified concentration
HCl (Titrant)	VWR	0.02 N

9.2. Alternately, you may prepare alkalinity standard and HCl standard solutions as follows:

9.2.1. 0.05N Na_2CO_3

9.2.1.1. Oven dry Na_2CO_3 at 250°C for at least 4 hours and cooled in dessicator.

9.2.1.2. Accurately weigh 2.5 ± 0.2 g and dissolve it in 1 liter reagent water.

9.2.1.3. Provide 7 days expiration date for this standard.

9.2.2. Titrant (Standard Acid)

9.2.2.1. HCL stock solution; 1N: dilute 8.33 ml conc. HCl to 100 ml reagent water

9.2.2.2. HCL standard solution; 0.02 N: dilute 20 ml of 1 N HCl to 1000 ml reagent water

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Aqueous Samples

10.1.1.1. Allow sample(s) to equilibrate at room temperature.

10.1.1.2. Transfer an appropriate amount of sample into a properly labeled container (see 10.4). Use reagent water for method blank and a certified standard for LCS/LCSD. Prepare a sample duplicate (SD) for every preparative batch. MS/MSD may also be included in the preparative batch depending on the project requirements.

10.1.1.3. The samples are now ready for analysis.

10.1.2. Soil/Solid Samples

10.1.2.1. Weigh 50 g of sample and add 50 ml of reagent water.

10.1.2.2. Place it in the shaker and agitate for 1 hour.

10.1.2.3. Centrifuge for 5 minutes.

10.1.2.4. Carefully, withdraw the leachate, and prepare them as described in 10.4.

10.2. **Instrument Parameters**

10.2.1. pH Meter

10.2.1.1. Electrode Channel - Channel 1

10.2.1.2. Resolution - 0.01 pH

10.2.2. Autotitrator

10.2.2.1. Electrode port - Port 1

10.3. **Calibration**

10.3.1. Manual Calibration

10.3.1.1. Transfer about 10 ml each of pH 4, 7 and 10 buffer solution into separate 20 ml vials and drop a piece of magnetic bar into each vials.

10.3.1.2. Rinse the pH meter electrode and shake off excess water.

10.3.1.3. Immerse electrode to the pH buffers and calibrate the pH meter, accordingly.

10.3.1.4. Record calibration readings in the analysis run log. Slope should be between 92- 102% to proceed to analysis.

10.3.2. Autotitration

10.3.2.1. Calibrations are performed by the autotitrator prior to sample analysis.

10.3.2.2. Transfer about 35 – 40 ml of pH 4, 7, 10 and 8 buffer solutions into separate 50 ml vials and load vials at locations 1, 2, 3 and 4 of sample tray, respectively.

10.3.2.3. After calibration is complete check if buffer solution 8 passes QC criteria of pH = 8 ± 0.05. If calibration fails to meet QC parameter, resequence and recalibrate.

10.4. **Analysis**

10.4.1. Standardization of Titrant

10.4.1.1. Titrants are purchased with certificate of analysis and concentration is verified with the analysis result of the LCS.

10.4.1.2. Perform triplicate standardization of titrant against Na_2CO_3 @ 0.05 N concentration when titrant is prepared in-house.

10.4.2. Manual Titration

10.4.2.1. **Initial pH Determination**

10.4.2.1.1. Place a clean magnetic stir bar in the sample. Position the sample container on the center of the magnetic stirrer. Switch the magnetic stirrer power on and gradually turn the speed knob until adequate mixing is obtained.

10.4.2.1.2. Rinse the calibrated pH probe with reagent water. Shake-off the excess water into a Kim wipes paper towel. Immerse the pH meter probe into the sample. Allow the meter readout to stabilize.

10.4.2.1.3. Record the pH reading on the initial pH column. Proceed to 10.4.2.2.

10.4.2.2. **Sample Titration**

10.4.2.2.1. Fill the burette with titrant up to the mark. Position the burette to the center of the flask.

10.4.2.2.2. Titrate an appropriate amount of of sample to pH = 4.5 (± 0.04) using 0.02N HCl. Record the titrant volume under pH=4.5 column. Results for total, bicarbonate, carbonate and hydroxide alkalinity are automatically calculated by the template. Alkalinity relationships/calculations are shown in Section 10.5.

10.4.2.3. **Sample Titration for Low Alkalinity**

10.4.2.3.1. For alkalinity of < 20 mg/L, titrate 100 ml of sample using 10 ml microburette an 0.02N acid solution.

10.4.2.3.2. Stop titration at pH range 4.5 ± 0.04. Record volume and exact pH.

10.4.2.3.3. Carefully, add titrant to lower the pH to exactly 0.3 pH units and record the titrant volume.

10.4.2.3.4. Record the actual final pH at the final pH column.

10.4.3. Automatic Titraton

10.4.3.1. **Program Setup**

10.4.3.1.1. Turn-on PC and activate PC Titrator program.

10.4.3.1.2. Click on “Titrator” located at the menu bar and access “manual control”

10.4.3.1.2.1. Click on autosampler tab to load autosampler tray template and home sampler.

10.4.3.1.2.2. Next, click on the digital tab to rinse the syringe and titration cell using pumps 2, 3, and 4.

Note: Pump 2: Turns on/off syringe pump

Pump 3: Turns on/off drain

Pump 4: Turns on/off rinse pump

10.4.3.1.2.3. Check for air bubbles in the burette. If air bubbles are found, click on the serial devices tab and dispense a few ml of titrant to push the bubble out. Refill burette and repeat if necessary.

10.4.3.1.2.4. Return to the menu screen when finished.

10.4.3.1.3. From the menu screen, click on "Titrator" and access "Run titration"

10.4.3.1.3.1. Load template "Daily run no order #" and input analytical batch sequence.

Note: Remember to include a sample duplicate for every preparative batch. ICV/CCV/MS/MSD may also be included in the preparative batch depending on the project requirements.

10.4.3.1.3.2. Check sequence for accuracy and completeness. Print and retain a copy of the timetable for reference.

10.4.3.2. Sample Titration

10.4.3.2.1. Aliquot 40 ml of sample into 50 ml vials and load samples as listed on the sequence.

10.4.3.2.2. Click on the "Start" button after loading pH buffers and samples. If the sequence needs to be updated during the analysis, click "priority" on the right to pause. DO NOT CLICK STOP! Afterwards, make the required changes and click on resume to continue onto the next sample.

Note: Load 40 ml of pH buffer 7/KCl probe solution at tray location 122 before the analytical batch is complete.

10.4.3.2.3. Print the report that appears on the screen when the run is completed. Close the screen and click OK to return to the main menu.

10.4.3.2.4. Click on "Titrator" and "Examine calibrations". Print the calibration record from the previous run by choosing "port 1" on the drop-down bar. Previous calibrations can also be checked by clicking the arrows at the bottom-left of the screen.

10.5. **Calculations**

10.5.1. Calculate for Titrant Concentration (if prepared in-house)

$$N = \frac{A \, x \, B}{53.00 \, x \, C} \qquad \text{Eq.-10.5.1}$$

where:

N = Normality of titrant

A = g Na_2CO_3 dissolved in 1 L solution

B = ml Na_2CO_3 solution titrated

C = ml titrant used to inflection point

10.5.2. Phenolphtalein Alkalinity (P)

$$P = \frac{A \, x \, C \, x \, 50000}{D} \qquad \text{Eq.-10.5.2}$$

10.5.3. Total Alkalinity (T)

$$T = \frac{B\,x\,C\,x\,50000}{D} \qquad \text{Eq.-10.5.3}$$

where:

A = volume of titrant added to reach pH 8.3

B = volume of titrant added to reach pH 4.5 (or 4.2 for low alkalinity)

C = concentration of titrant, normality

D = volume of sample used

Alkalinity Relationships/Calculations			
Result of Titration	Hydroxide (OH) Alkalinity as $CaCO_3$	Carbonate (CO_3) Alkalinity as $CaCO_3$	Bicarbonate (HCO_3) Alkalinity as $CaCO_3$
P = 0	0	0	T
P < ½ T	0	2P	T – 2P
P = ½ T	0	2P	0
P > ½ T	2P - T	2 (T-P)	0
P = T	T	0	0

where:

P = phenolphthalein alkalinity

T = total alkalinity

10.5.4. Percent Recovery

$$R = \left(\frac{C_f - C_s}{C_o}\right) \times 100 \qquad \text{Eq.-10.5.4}$$

where:

R = percent recovery

C_f = found concentration

C_s = sample concentration (for LCS, $C_s = 0$)

C_o = known spike concentration

10.5.5. Relative Percent Difference

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.5}$$

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 (if applicable)

10.6.2. Print a copy of the raw data and the QC report.

10.6.3. Highlight reportable results for data processing.

10.7. **Report Generation**

10.7.1. Manual Titration Report

10.7.1.1. Generate the .txt file using WDB5NT1.exe.

10.7.1.2. Generate the sample result using MSRB6T2.exe.

10.7.1.3. Generate the LCS/LCSD summary using QCICT3N1.exe.

10.7.1.4. Generate the sample duplicate result using CQITN1.exe.

10.7.1.5. Generate the case narrative using CN2.exe.

10.7.2. Automatic Titration Report

10.7.2.1. After analysis, access “Reporting” from the main menu and click on “Print or prepare shazam reports”. Open report format “Water Analysis Historical Data Report.srw”. Double-click on the filters and input the run # found on the front of the report printout. Export the data in an ASCII delimited text format onto the desktop by clicking on “File” and then “Export” from the menu bar. Transfer the export data onto the network via a flash drive for reporting.

10.7.2.2. Convert exported data file for reporting using ALKALN.exe.

10.7.2.3. Generate the .txt file using WDB5NT.exe.

10.7.2.4. Generate the sample result using MSRB6T.exe.

10.7.2.5. Generate the LCS/LCSD summary using QCICT3A.exe.

10.7.2.6. Generate the sample duplicate result using CQIT.exe.

10.7.2.7. Generate the case narrative using CN2.exe.

10.8. **Data Review**

10.8.1. Arrange the analysis package in sequence as detailed below. Attach all raw data to every form generated, to include re-analysis.

- Case Narrative
- Sample Results
- LCS/LCSD Summary
- Sample Duplicate Summary
- Analytical Run Log
- Raw Data
- Non-Conformance Report (If any)

10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

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. The pH electrodes should be maintained clean and the probe returned to the probe handle after every use.

10.9.2. Instruments should receive routine preventive maintenance, which is reported in instrument-specific maintenance logs. Routine maintenance ensures that all equipment are operating under optimum condition, thus reducing the possibility of instrument malfunction that consequently affects sample results.

11.0 QUALITY CONTROL

11.1. Refer to Appendix 1 for all related Quality Control parameters, frequency, acceptance criteria and corrective action.

11.2. All labwares used in the sample preparation shall be properly treated as specified in EMAX-QC07.

11.3. All analytical standards are prepared fresh on the day of analysis. All analytical standard preparations shall follow the procedures described in EMAX-QC02 and shall be documented in the analytical standard preparation log.

11.4. Instrument parameters are checked and recorded in the instrument maintenance log prior to its use for the day.

11.5. A preparative batch shall include a MB, LCS, SD (sample duplicate) and a maximum of 20 field original samples unless otherwise specified by the project.

11.6. A valid LOQ must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.7. 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 action for each Quality Control procedure is summarized in Appendix 1.

12.2. QC Samples

12.2.1. Method Blank (MB). If MB is non-compliant consider the following suggestions to analyze and possibly correct the problem:

- Investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.
- If the analyte found in the 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-extract and re-analyze the method blank with the associated samples.

12.2.2. Lab Control Sample(s). If LCS is non-compliant consider the following suggestions to analyze and possibly correct the problem:

- Check for errors in calculation and concentration of the analyte solution
- If recovery of LCS is bias-high and associated sample results are non-detect, then results can be reported. If associated samples show positive results or if recovery of LCS is bias-low, then acquire or prepare a new LCS standard and titrant and re-analyze with the associated samples.
- If recovery of LCS is still non-compliant after re-analysis, check the PSR for corrective action otherwise; discuss the issue in the case narrative.

12.3. If pH electrode response is erratic and/or sluggish, record sample description on the analytical log and inform the Supervisor for further advice.

12.4. For insufficiency of sample(s), inform the Supervisor immediately for further advice.

12.5. Other anomalies encountered during the analytical process not listed in Appendix 1 shall require a non-conformance report (NCR). Refer to EMAX-QA08.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of sample that may go to the wastewater drains.

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. Separate and properly identify all unused expired analytical standards for proper disposal.

14.3. No sample shall be dumped in the laboratory sink.

14.4. Dispose all unused samples, expired analytical standards and other wastes generated during the analytical process in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

15.1.1. Alkalinity of water – is its acid-neutralizing capacity. It is the sum of all titratable bases.

15.1.2. Phenolphthalein Alkalinity – is the alkalinity measured by titration to pH 8.3

1.1.1. 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.

15.1.2.1. **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.

15.1.3. 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.

15.1.4. Material Safety Data Sheet (MSDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.5. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.6. Reagent Water – is purified water free from any target analyte or any other substance that may interfere with the analytical process.

15.1.7. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

15.1.8. 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.

15.1.9. 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.

15.1.10. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.11. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.12. Matrix – is a component or form of a sample.

15.1.13. Re-analysis – is a repeated analysis from the same extract/leachate or sample, identified with the Lab Sample ID suffixed with “W”.

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 analysis for Alkalinity. 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.

15.4. **Department of Energy Basic Ordering Agreement (DoE-BoA) 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. Method 2320B, Standard Methods for the Examination of Water and Wastewater, 20th Edition.

16.2. Method 310.1, EPA Methods for Chemical Analysis of Water and Wastes, Editorial Rev. 1978.

16.3. 40 CFR Federal Register, Rules and Regulations Vol. 12, No.27, p. 11236, March 2007.

16.4. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES

17.1. **Figures**

17.1.1.	Figure 1	Typical Raw Data
17.1.2.	Figure 1a	Typical Raw Data for Autotitration
17.1.3.	Figure 2	Typical Sample Result Summary
17.1.4.	Figure 3	Typical LCS/LCSD Summary
17.1.5.	Figure 4	Typical Sample Duplicate Result Summary
17.1.6.	Figure 5	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.	2320BFA	Analytical Run Log
17.3.2.	2320BFM	Instrument Maintenance Log

Figure 1: **TYPICAL RAW DATA**

EMAX LABORATORIES, INC.

ALG003.xls

ALKALINITY RAW DATA

MISCELLANEOUS INFORMATION		
Standard	Concentration	
Acid Titrant, HCl	0.02	N
Na_2CO_3 (Zst Z_N)*	2.63	g/L
LCS	399	mg/L
MS	500	mg/L
MDL	5	mg/L
RL	5	mg/L

pH Meter Calibration			
Instrument ID:		58	
Buffer 4	4.01	07/08/11	15:10:26
Buffer 7	7	07/08/11	15:11:21
Buffer 10	10.03	07/08/11	15:12:16
pH Check Buffer (8)	7.97	07/08/11	15:13:48

$N = \frac{(Z)(V_{Na_2CO_3})}{(53)(V_{HCl})}$ $N = \frac{(Z)(N)(V_{Na_2CO_3})}{(V_{HCl})}$

* Standardization of Acid Titrant(N)
Z=g/L Na_2CO_3. If the concentration of Na_2CO_3 is in N, then $Z=Z_N$.

STANDARDIZATION				
$V_{Na_2CO_3}$ (L) (ml)	V_{HCl} (ml)	DATE	TIME	Acid Titrant HCl (N)
				NA
				#DIV/0!
				#DIV/0!
				#DIV/0!
			Average HCl (N)=	0.02000

* Acid Titrant is purchased as certified standard solution

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SOP: EMAX-2320B Rev. 0

Analytical Batch: **ALG003**

Analyzed By: RC

Data File Name	Lab Sample ID	Sample Amount (ml)	Sample pH	Analysis Date mm/dd/yy	Analysis Time hh:mm:ss	Volume of Acid Titrant (A) ml pH=8.3	Analysis Date mm/dd/yy	Analysis Time hh:mm:ss	Volume of Acid Titrant (B) ml pH=4.5	Analysis Date mm/dd/yy	Analysis Time hh:mm:ss	Vol.of Acid Titrant (C) ml pH=4.2	Analysis Date mm/dd/yy	Analysis Time hh:mm:ss	Final pH	Analysis Date mm/dd/yy	Analysis Time hh:mm:ss	NOTES
ALG003-01	ALG003WB	100	5.47	07/08/11	15:16:41	NA	07/08/11	15:16:45	0.13	07/08/11	15:17:21	0.25	07/08/11	15:17:55	4.19	07/08/11	15:18:00	
ALG003-02	ALG003WL	5	9.1	07/08/11	15:19:43	1.33	07/08/11	15:22:10	2.01	07/08/11	15:23:06	NA	07/08/11	15:23:12	4.49	07/08/11	15:23:15	
ALG003-03	ALG003WC	5	9.11	07/08/11	15:26:43	1.3	07/08/11	15:28:49	2	07/08/11	15:30:26	NA	07/08/11	15:30:28	4.51	07/08/11	15:30:31	
ALG003-04	F242-01	2	6.91	07/08/11	15:31:23	NA	07/08/11	15:31:25	1.16	07/08/11	15:34:36	NA	07/08/11	15:34:38	4.49	07/08/11	15:34:39	
ALG003-05	F242-01D	2	6.81	07/08/11	15:37:53	NA	07/08/11	15:40:13	1.14	07/08/11	15:40:15	NA	07/08/11	15:40:16	4.54	07/08/11	15:40:18	
ALG003-06	F242-01M	2	8.75	07/08/11	15:45:53	0.12	07/08/11	15:47:01	1.57	07/08/11	16:02:29	NA	07/08/11	16:02:30	4.51	07/08/11	16:02:32	
ALG003-07	F242-02	2	6.77	07/08/11	16:05:44	NA	07/08/11	16:05:47	1.82	07/08/11	16:13:54	NA	07/08/11	16:13:57	4.48	07/08/11	16:13:59	
ALG003-08	F242-03	100	5.38	07/08/11	16:19:09	NA	07/08/11	16:19:14	0.12	07/08/11	16:20:49	0.25	07/08/11	16:21:32	4.24	07/08/11	16:21:34	
ALG003-09	F248-02	2	6.94	07/08/11	16:24:54	NA	07/08/11	16:24:56	1.51	07/08/11	16:29:48	NA	07/08/11	16:29:50	4.47	07/08/11	16:30:00	
ALG003-10	F248-03	2	6.59	07/08/11	16:34:44	NA	07/08/11	16:35:15	1.45	07/08/11	16:36:38	NA	07/08/11	16:36:39	4.46	07/08/11	16:36:42	
ALG003-11	F248-04	100	5.17	07/08/11	16:39:22	NA	07/08/11	16:39:23	0.1	07/08/11	16:40:10	0.21	07/08/11	16:40:39	4.22	07/08/11	16:41:02	
ALG003-12	F248-05	100	5.49	07/08/11	16:44:11	NA	07/08/11	16:44:14	0.15	07/08/11	16:46:27	0.3	07/08/11	16:46:31	4.21	07/08/11	16:46:33	
ALG003-13	F248-06	2	6.93	07/08/11	16:50:52	NA	07/08/11	16:50:54	2.19	07/08/11	16:54:30	NA	07/08/11	16:54:32	4.52	07/08/11	16:54:38	

Figure 1a: **TYPICAL RAW DATA FOR AUTOTITRATION**

Report Date: 09/01/2011 : 12:26 PM

PC-TitratION PLUS

pH and Alkalinity In-Run Report

Run Number 528 Analytical Batch# 11E5I01

Seq. #	SampleID	Data File Name	RunDate	RunTime	pH	Volume @pH 8.3	Volume @pH 4.5	Volume @pH 4.2	palk-ppm	talk-ppm	bcarb-ppm	carb-ppm	hydr-ppm	Review Notes
1	ICV PH8	11E5I01	09/01/2011	10:39 AM	7.99	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	-1.00	
2	RINSE	11E5I01	09/01/2011	10:44 AM	4.99	.00	.02	.03	.00	.56	.56	.00	.00	
3	ALI001WB	11E5I01	09/01/2011	10:48 AM	4.09	.00	.00	.00	.00	.00	.00	.00	.00	
4	ALI001WL	11E5I01	09/01/2011	11:03 AM	8.80	4.74	9.99	-1.00	189.45	399.50	20.61	378.89	.00	
5	ALI001WC	11E5I01	09/01/2011	11:11 AM	8.84	5.01	10.00	-1.00	200.34	399.89	.00	399.11	.78	
6	H838-01	11E5I01	09/01/2011	11:20 AM	7.51	.00	5.00	-1.00	.00	199.82	199.82	.00	.00	
7	H838-01D	11E5I01	09/01/2011	11:27 AM	7.60	.00	4.92	-1.00	.00	196.89	196.89	.00	.00	
8	H838-02	11E5I01	09/01/2011	11:34 AM	7.67	.00	4.89	-1.00	.00	195.80	195.80	.00	.00	
9	H838-03	11E5I01	09/01/2011	11:41 AM	7.74	.00	5.00	-1.00	.00	199.94	199.94	.00	.00	
10	H838-04	11E5I01	09/01/2011	11:48 AM	7.73	.00	5.01	-1.00	.00	200.24	200.24	.00	.00	
11	H838-05	11E5I01	09/01/2011	11:57 AM	7.76	.00	5.02	-1.00	.00	200.71	200.71	.00	.00	
12	H838-06	11E5I01	09/01/2011	12:04 PM	7.79	.00	4.98	-1.00	.00	199.07	199.07	.00	.00	
13	H838-07	11E5I01	09/01/2011	12:11 PM	7.77	.00	5.10	-1.00	.00	203.92	203.92	.00	.00	
14	H838-08	11E5I01	09/01/2011	12:18 PM	7.78	.00	5.10	-1.00	.00	204.16	204.16	.00	.00	
15	RINSE	11E5I01	09/01/2011	12:24 PM	5.61	.00	.04	.09	.00	-.34	-.34	.00	.00	

Analyzed by: Rc Reviewed by: Rc Date: 9/1/11

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PC-TitratION PLUS by Man-Tech Associates, Inc.

pH AND ALKALINITY IN-RUN REPORT.SRW
Copyright Hinterland Software Ltd.

Figure 2: TYPICAL SAMPLE RESULT SUMMARY

METHOD 2320B
ALKALINITY

Client : XYZ, INC.
Project : CLEAN WATER PROJECT
Batch No. : 11F242

Matrix : WATER
Instrument ID : 53

SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	DLF	MOIST	RL (mg/L)	MDL (mg/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK1W	ALG003WB	ND	1	NA	5.00	5.00	07/08/1115:18	NA	ALG003-01	NA	ALG003W	NA	NA
LCS1W	ALG003WL	402	1	NA	5.00	5.00	07/08/1115:23	NA	ALG003-02	NA	ALG003W	NA	NA
LCD1W	ALG003WC	400	1	NA	5.00	5.00	07/08/1115:30	NA	ALG003-03	NA	ALG003W	NA	NA
25GW114-0611	F242-01	1160	1	NA	5.00	5.00	07/08/1115:34	NA	ALG003-04	NA	ALG003W	06/28/1112:33	06/29/11
25GW114-0611DUP	F242-01D	1140	1	NA	5.00	5.00	07/08/1115:40	NA	ALG003-05	NA	ALG003W	06/28/1112:33	06/29/11
25GW121-0611	F242-02	1620	1	NA	5.00	5.00	07/08/1116:13	NA	ALG003-07	NA	ALG003W	06/28/1110:33	06/29/11
25GW129-0611	F242-03	ND	1	NA	5.00	5.00	07/08/1116:21	NA	ALG003-08	NA	ALG003W	06/28/1114:40	06/29/11

Figure 3: TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 11F242
METHOD: METHOD 2320B

MATRIX: WATER
% MOISTURE: NA

DILUTION FACTOR:	1	1	1
SAMPLE ID:	MBLK1W		
LAB SAMP ID:	ALG003WB	ALG003WL	ALG003WC
LAB FILE ID:	ALG003-01	ALG003-02	ALG003-03
DATE EXTRACTED:	NA	NA	NA
DATE ANALYZED:	07/08/1115:18	07/08/1115:23	07/08/1115:30
PREP. BATCH:	ALG003W	ALG003W	ALG003W
CALIB. REF:	NA	NA	NA

DATE COLLECTED: NA
DATE RECEIVED: NA

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 (%)
Alkalinity	ND	399	402	101	399	400	100	0	80-120	20

Figure 4: **TYPICAL SAMPLE DUPLICATE SUMMARY**

EMAX QUALITY CONTROL DATA
DUPLICATE SAMPLE ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 11F242
METHOD: METHOD 2320B

MATRIX:	WATER		% MOISTURE:	NA
DILUTION FACTOR:	1	1		
SAMPLE ID:	25GW114-0611			
EMAX SAMP ID:	F242-01	F242-01D		
LAB FILE ID:	ALG003-04	ALG003-05		
DATE EXTRACTED:	NA	NA	DATE COLLECTED:	06/28/11 12:33
DATE ANALYZED:	07/08/1115:34	07/08/1115:40	DATE RECEIVED:	06/29/11
PREP. BATCH:	ALG003W	ALG003W		
CALIB. REF:	NA	NA		

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	DUPL RSLT (mg/L)	RPD RSLT %	QC LIMIT (%)
Alkalinity	1160	1140	2	20

Figure 5: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER PROJECT

SDG : 11F242

METHOD 2320B
ALKALINITY

A total of three (3) water samples were received on 06/29/11 for Alkalinity analysis, Method 2320B in accordance with Standard Methods for the Examination of Water and Wastewater, 20th Edition.

Holding Time
Samples were analyzed within the prescribed holding time.

Calibration
Calibration was performed as prescribed by the method. 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 ALG003WL/C were all within QC limits.

Matrix QC Sample
Sample duplicate was 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 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Instrument Calibration	Daily prior to sample analysis	± 0.05 pH units	Correct the problem and re-calibrate prior to sample analysis		
Method Blank	One method blank per preparation batch	Result ≤ LOQ	If sample results are non-detect, no corrective action is needed, otherwise correct the problem and re-analyze all associated samples.		
LCS	One LCS per preparation batch	% Recovery: 80 – 120%	Correct the problem and re-analyze all associated samples.		
Sample Duplicate	One sample duplicate per preparation batch	RPD ≤ 20%	Discuss in the case narrative		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
20th Edition			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
METHOD SM 2320B

Unit: mg/L
Sample Amount(ml): 25

Analyzed By: R. Cheung
Date Analyzed: 09/02/11
09/09/11

PARAMETER	E5I02004 ALI002WL	E5I02005 ALI002WC	E5I03004 ALI003WL	E5I03005 ALI003WC	True Value (mg/L)	Ave Conc.	Ave % Rec.	SD	RPD	QC Criteria	Comments
Alkalinity	394	394	402	401	399	398	100	4.349	1.09	80 - 120	Passed

2320BFA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
ALKALINITY

Page 1

Note: For samples and relevent QCs/Standards analyzed, refer to attached analytical sequence.

☐ Titration end point: pH 4.5 ± 0.04

☐ Low alkalinity: pH 4.2 ± 0.04

MS/MSD amount of spike:

Comments:

Book#: AAL-031

Instrument No.: ☐ 53 ☐ 97 ☐ E5

Analytical Batch:

Analytical Sequence:

SOP #	Rev. #
☐ EMAX-310.1	
☐ EMAX-2320B	
☐ EMAX-	

STANDARDS ID	CONC. (mg/L)
0.02N HCL	
LCS	
MS/MSD	
ICV	

pH Buffer	ID
pH 4	
pH 7	
pH 10	
pH 8.0 (Check)	

Analyzed By:

Date:

2320BFM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: E5 Month: Year: 201_ Book # ME5-002

Date							
Titrant Bottle Level							
DI Bottle Level							
Waste Bottle Level							
Titration Module							
Buret Module							
pH Probe							
Printer Paper							
Autosampler							
Analyst Initials							
Trouble Shooting							
Problem(s) Encountered							
Corrective Action							
Return To Spec (Analyst Initials)							

Code	Problems Encountered	Code	Corrective Action

SOP QA REVIEW FORM

EMAX-2540C	Rev. 8	TOTAL DISSOLVED SOLIDS
SOP No.	Revision Number	Title

SECTION	✓	COMMENTS
Scope & Application	✓	
Summary of Method	✓	
Detection Limits	✓	
Dynamic Range	✓	
Sample Holding Time and Preservation	✓	
Associated SOPs	✓	
Safety	✓	
Instrument, 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 necessary.

Reviewed By FARINA MADAMBA Date: 5/17/16

STANDARD OPERATING PROCEDURES

TOTAL DISSOLVED SOLIDS

SOP No.:	EMAX-2540C	Revision No.	8	Effective Date:	12-Mar-15
Prepared By:	Mary Jane Mendoza			Date:	03.12.15
Approved By:	Kenette Pimentel QA Manager			Date:	03.12.15
Approved By:	Caspar Pang Laboratory Director			Date:	03-12-15

Control Number: 2540C-08-

1.0 SCOPE AND APPLICATION

1.1. This method describes the determination of total dissolved solids (TDS) in drinking water, groundwater samples and other aqueous samples of similar matrix.

1.2. This SOP is an adaptation of Method 2540C-1997, Standard Methods for the Examination of Water and Wastewater, 20th and 21st Editions.

2.0 SUMMARY OF METHOD

2.1. A well-mixed required amount of sample is filtered through a glass fiber filter. The filtrates are collected into a pre-weighed evaporating dish, pre-heated at 90°C and continue drying to a constant weight at 180°C. The increase of weight in the dish represents the total dissolved solids.

2.2. **Interferences**

2.2.1. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

2.2.2. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.

2.2.3. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue must be limited to about 200 mg.

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL) & Limit of Detection (LOD)**

3.1.1. DL and LOD are not applicable for this method.

3.2. **Limit of Quantitation (LOQ)**

3.2.1. Refer to EMAX-QA04 for validation, verification and generation of LOQ.

3.2.2. Established LOQ for this method is 10 mg/L.

4.0 DYNAMIC RANGE

4.1. 2.5 mg – 200 mg dried residue

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Sample Collection**

5.1.1. Samples received in the laboratory are expected to be contained in HDPE bottles and cooled to ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. Analyze samples within seven days from collection date.

5.3. **Preservation**

5.3.1. Store the samples at ≤ 6°C without freezing.

6.0 ASSOCIATED SOPs

6.1. EMAX-160.1 Total Dissolved Solids

6.2. EMAX-DM01 Data Flow and Review

6.3. EMAX-QA04 Detection Limit (DL)

6.4. EMAX-QA05 Training

6.5. EMAX-QA08 Corrective Action

6.6. EMAX-QC01 Quality Control for Chemicals

6.7. EMAX-QC04 Balance Calibration

6.8. EMAX-QC06 Volumetric Labware and Micropipette Verification

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 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.

7.3. If for any reason, reagents and/or samples get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with copious amount of tap 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 AND REAGENTS

8.1. **Instruments and Supplies**

Filtration assembly	SCP Science DigiFilter Manifold, 12 position or equivalent

Filters	SCP Science DigiFilter 0.45 micron filters or equivalent
Graduation cylinder	Class A 100 ml, 25 ml, 10 ml
Evaporating Vessel	20, 40 ml scintillation vial, 150 ml beaker
Micropipette	Variable
Wash bottles	Plastic
Oven	Fisher Isotemp oven, temperature range: 20°C – 200°C or equivalent
Balance	Sartorius H-51 accurate to 0.0001 g, or equivalent
Weights	Class "S"; ASTM1 certified; 0.1g, 5g, 100g
Desiccator	With color indicator dessicant
DigiTubes	50 ml or equivalent

8.2. **Chemicals and Reagents**

8.2.1. Reagent Water – Type II

9.0 STANDARDS

9.1. Sodium Chloride Standard (LCS) 1000 mg/L – Weigh 1 g of sodium chloride (previously dried for at least 1 hour). Dissolve in reagent water and dilute to 1000 ml.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Preparation for Filtration

10.1.1.1. Connect the Digifilter to a Digitube and attach to the DigiFilter Manifold.

10.1.1.2. Apply vacuum and rinse the filter with three successive portions of 10 ml reagent water.

10.1.1.3. Continue to suction until no trace of water is evident.

10.1.2. Prepare Evaporating Vessel

10.1.2.1. Heat clean the vials at 180°C (± 2°C) for 1 hour.

10.1.2.2. Cool it in the dessicator until needed.

10.1.2.3. Weigh before use.

10.1.3. Preparative Batch

10.1.3.1. Bring sample to room temperature.

10.1.3.2. Estimate the amount of sample to be used in the analysis by checking the conductivity of the samples.

- If conductivity is below 800 µmho/cm, use 100 ml of sample.

- If conductivity is about 800 – 10000 µmho/cm, use 50 ml of sample.
- If conductivity is about 1000 – 5000 µmho/cm, use 20 ml of sample.
- If conductivity is about 5000 – 10000 µmho/cm, use 10 ml of sample.
- If conductivity is about 10000 – 25000 µmho/cm, use 5 ml of sample.
- If conductivity is about 25000 – 70000 µmho/cm, use 2 ml of sample.
- If conductivity is greater than 70000 µmho/cm, please check with supervisor.

Historical information of samples and appearance can also help in estimating the amount of sample to be used.

10.1.3.3. For a maximum of 20 field samples, prepare one method blank using reagent water, one LCS (refer to Section 9.0) and a sample duplicate for every 10 samples.

10.2. **Instrument Parameters**

10.2.1. Set the oven to deliver 90°C (± 5°C) for preheating and 180°C (± 2°C) for drying. Record the thermometer ID in the analytical log.

10.3. **Calibration**

10.3.1. Calibrate the analytical balance at 0.1 g, 5 g and 100 g using class "S" weights. Record the balance and weight ID.

10.3.2. Calibration must be within ± 0.1% or 0.5mg, whichever is higher.[1]

10.3.3. Verify calibration of the balance before and after each analytical batch. If calibration is not verified after the measurements, re-calibrate the balance and re-weigh the batch.

10.4. **Analysis**

10.4.1. TDS Determination

10.4.1.1. Shake the sample thoroughly and volumetrically measure the amount of sample for analysis as determined in Section 10.1.3.2. If historical information are available suggesting that sample is of known high TDS concentration or if sample appears to be turbid or cloudy, consider lesser amount of sample for analysis.

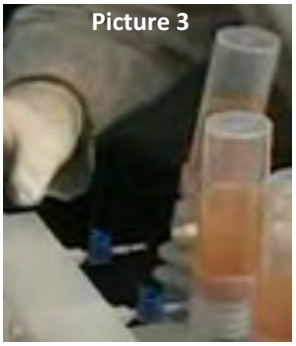
10.4.1.2. Pour the sample into a graduated cylinder and transfer into a labeled DigiTube. Rinse the graduated cylinder with reagent water and transfer the rinsate to the DigiTube (see picture 1).

10.4.1.3. Connect the DigiTube to the pre-washed DigiFilter connected to another DigiTube which serves as receiver for the filtrate (see Picture 2). Record DigiFilter Lot Number in the Run Log.

[1] Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, version 5.0, Module 2 page 26.

10.4.1.4. Invert the DigiTube assembly so that your sample is on top and connect it to the vacuum manifold (see Picture 3). Turn the vacuum pump on and open the Luer lock dial to the open position to allow the sample to be pulled through the digiFilter (see Picture 4).

10.4.1.5. Unscrew the emptied DigiTube and rinse it with two portions of 5 ml reagent water using a reagent water wash bottle. Transfer each washing to the DigiFilter. Give the filter a final rinse of approximately 5 ml using a reagent water wash bottle.


Picture 3

10.4.1.6. Continue suction until no trace of water is evident.

10.4.1.7. Turn off the vacuum to release excess pressure.

10.4.1.8. Transfer the filtrate with the rinsate into a weighed evaporating vessel.

10.4.1.9. Place the sample vessel in the oven (set at 90°C) and evaporate until sample is at most dry. If evaporating vessel can not contain the entire sample at one time, add the successive portions on the same dish used for evaporation.

Picture 4

10.4.1.10. Dry evaporated sample for at least one hour in an oven at 180°C (± 2°C).

10.4.1.11. After the sample has been completely dried, cool the sample in a desiccator and weigh. Check if dried residue is between 2.5 mg to 200 mg.

- If dried residue is < 2.5 mg and sample aliquot volume used satisfies LOQ requirement, report result. Otherwise, repeat sample analysis using higher sample aliquot volume up to 1 liter to ensure that representative sub-sample is taken.
- If residue is > 200 mg, repeat analysis using lesser volume of sample estimated from the result.

10.4.1.12. Repeat drying process at 180°C (± 2°C) until weight is constant or until weight loss is less than 0.0005 g.

10.4.2. Sample Result Evaluation

10.4.2.1. Check the QC data as soon as available.

- ✓ Check the method blank and make sure that it meets the project requirement.
- ✓ Check the LCS and make sure it meets the recovery limit.
- ✓ Check the sample duplicate(s) that it meets the acceptance criteria or note possible matrix interference on the run log.
- ✓ Check that all samples are analyzed within the dynamic range.

10.4.2.2. Report only samples that are properly quantitated. (Samples between 2.5 – 200 mg per sample aliquot volume that satisfies LOQ requirement).

10.5. **Calculations**

10.5.1. Calculate for Total Dissolved Solids (TDS)

$$TDS, mg/L = \frac{(R-D)\times 1000}{S} \quad \text{Eq-10.5.1}$$

where:

R – Weight of residue + dish, mg

D – Weight of dish, mg

S – Volume of sample analyzed, ml

10.5.2. Calculate for Percent Recovery

$$\%Recovery = \frac{(C_f - C)}{C_s} \times 100 \quad \text{Eq-10.5.2}$$

where:

C_f – Concentration found

C – Concentration of the sample (use 0 for LCS)

C_s – Concentration of spike

10.5.3. Calculate for Precision

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C1 + C2}{2}\right)} x100 \quad \text{Eq-10.5.3}$$

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. Print a copy of the analytical run log.

10.6.2. Highlight the data to be reported.

10.6.3. 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, sample results and QC summaries using GRAV.exe.

10.7.2. Generate the case narrative using CN00.exe.

10.7.3. Arrange the analysis package in sequence as detailed below.

10.7.3.1. Case Narrative

10.7.3.2. Sample Results

10.7.3.3. LCS/LCSD Summary

10.7.3.4. Sample Duplicate Result Summary

10.7.3.5. Run Log

10.7.3.6. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01and the PSR.

10.8.1.1. If any of the checkpoints below is non-compliant, re-analysis is required.

- ✓ Check that MB is compliant to PSR.
- ✓ Check LCS/LCSD recoveries against project specific criteria (PSR). In the absence of the PSR, default to in-house QC limits.
- ✓ Check that all sample results are within dynamic range.

10.8.1.2. Review the generated reports against the analytical run log. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.8.1.3. 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.

10.8.2. Submit the analytical folder for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Check the vacuum pump oil level prior to use.

10.9.2. Clean filter assembly thoroughly after each use.

11.0 QUALITY CONTROL

11.1. Acceptance criteria for each quality control procedure are summarized in Appendix 1.

11.2. **Analytical Batch QC**

11.2.1. Glass-fiber filters must be properly treated prior to use.

11.2.2. Filter assembly must be thoroughly cleaned prior to use.

11.2.3. Balance must be calibrated before and after each weighing.

11.2.4. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06 for details.

11.2.5. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.3. **Preparation Batch QC**

11.3.1. The maximum number of original field samples in a prep batch is 20 unless otherwise specified by the project.

11.3.2. Prepare method blank, LCS, and sample duplicate(s) in every prep batch, unless otherwise specified by the project. They are subjected to the same process that the field samples undergo as described in Section 10.

11.3.3. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.4. **Method QC**

11.4.1. A valid LOQ must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.4.2. All analysts conducting this analysis must demonstrate capability (DOC) as described in EMAX-QA05.

12.0 CORRECTIVE ACTION

12.1. Corrective action for each quality control procedure is summarized in Appendix 1.

12.2. **Preparation Batch QC**

12.2.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.2.2. When method blank is non-compliant, investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.

12.2.3. If LCS is non-compliant, investigate the source of the problem to institute resolution to correct, minimize or eliminate the problem.

12.2.4. If sample duplicate is non-compliant, discuss in case narrative.

12.3. 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.

12.3.1. Refer to EMAX-QA08 for NCR details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of standards and reagents that may go to the wastewater drains.

13.2. Prepare all standards in the fume hood.

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 wastes generated during analytical process in properly labeled satellite waste containers for proper disposal

14.4. Dispose all unused samples, expired analytical standards and other wastes 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.

15.1.1.2. **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.

15.1.2. 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.

15.1.3. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.4. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.5. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

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. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.8. 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.

15.1.9. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.10. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.11. Matrix – is a component or form of a sample.

15.1.12. Reagent Water – is purified water free from any target analyte or any other substances that may interfere with the analytical process.

15.2. Application of QC Procedures

15.2.1. The procedures and QC criteria summarized in this SOP applies to all projects when performing Total Dissolved Solids. 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 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, is applied.

16.0 REFERENCES

16.1. Standard Methods for the Examination of Water and Waste Water, 20th and 21st Editions, Method 2540C-1997

16.2. Title 40 Code of Federal Regulations, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, latest edition

16.3. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, version 5.0.

16.4. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Figures**

17.1.1. Figure 1 Typical Sample Result Summary

17.1.2. Figure 2 Typical LCS Summary

17.1.3. Figure 3 Typical Sample Duplicate Result Summary

17.1.4. Figure 4 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. Form 2540CFA Analytical Run Log

Figure 1: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD 2540C
TOTAL DISSOLVED SOLIDS

Client : XYZ, INC.
Project : CLEAN WATER
Batch No. : 15B148
Matrix : WATER
InstrumentID : 402426

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	PREP FACTOR	MOIST (%)	LOQ (mg/L)	MDL (mg/L)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
MBLK1W	TDB009WB	ND	1	NA	10	10	02/27/1517:01	NA	15TDB00901	15TDB009	TDB009W	NA	NA
LCS1W	TDB009WL	1030	1	NA	10	10	02/27/1517:01	NA	15TDB00902	15TDB009	TDB009W	NA	NA
14W-08B	B148-02	1820	1	NA	10	10	02/27/1517:01	NA	15TDB00903	15TDB009	TDB009W	02/26/1514:15	02/27/15
14W-08BDUP	B148-02D	1800	1	NA	10	10	02/27/1517:01	NA	15TDB00904	15TDB009	TDB009W	02/26/1514:15	02/27/15
14W-09	B148-03	1490	1	NA	10	10	02/27/1517:01	NA	15TDB00905	15TDB009	TDB009W	02/26/1515:30	02/27/15
14W-04	B148-04	1720	1	NA	10	10	02/27/1517:01	NA	15TDB00907	15TDB009	TDB009W	02/27/1510:10	02/27/15
14W-05	B148-05	1700	1	NA	10	10	02/27/1517:01	NA	15TDB00908	15TDB009	TDB009W	02/27/1510:15	02/27/15
14W-10	B148-06	1510	1	NA	10	10	02/27/1517:01	NA	15TDB00909	15TDB009	TDB009W	02/27/1510:40	02/27/15
14W-02	B148-07	1720	1	NA	10	10	02/27/1517:01	NA	15TDB00910	15TDB009	TDB009W	02/27/1511:25	02/27/15

Figure 2: TYPICAL LCS SUMMARY

EMAX QUALITY CONTROL DATA
LCS ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 15B148
METHOD : SM 2540C

MATRIX	WATER	
DILUTION FACTOR	1	1
SAMPLE ID	MBLK1W	LCS1W
LAB SAMPLE ID	TDB009WB	TDB009WL
LAB FILE ID	15TDB00901	15TDB00902
DATE PREPARED	NA	NA
DATE ANALYZED	02/27/1517:01	02/27/1517:01
PREP BATCH	TDB009W	TDB009W
CALIBRATION REF	15TDB009	15TDB009

ACCESSION:

PARAMETER	MB RESULT (mg/L)	SPIKE AMT (mg/L)	BS RESULT (mg/L)	BS REC (%)	QC LIMIT (%)
TDS	ND	1000	1030	103	80-120

Figure 3: TYPICAL SAMPLE DUPLICATE RESULT SUMMARY

EMAX QUALITY CONTROL DATA
SAMPLE DUPLICATE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 15B148
METHOD : SM 2540C

MATRIX	WATER	
DILUTION FACTOR	1	1
SAMPLE ID	14W-08B	14W-08BDUP
LAB SAMPLE ID	B148-02	B148-02D
LAB FILE ID	15TDB00903	15TDB00904
DATE PREPARED	NA	NA
DATE ANALYZED	02/27/1517:01	02/27/1517:01
PREP BATCH	TDB009W	TDB009W
CALIBRATION REF	15TDB009	15TDB009

ACCESSION:

PARAMETER	PARENT RESULT (mg/L)	DUP RESULT (mg/L)	RPD (%)	MAX RPD (%)
TDS	1820	1800	1	20

Figure 4: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN WATER

SDG : 15B148

METHOD SM 2540C
TOTAL DISSOLVED SOLIDS

A total of six(6) water samples were received on 02/27/15 for Total Dissolved Solids BY SM 2540C in accordance with Standard Methods for the Examination of Water and Wastewater, 21st Edition.

Holding Time
Samples were extracted and analyzed within the prescribed holding time.

Calibration
Balance calibration verifications were carried on a frequency specified by the method. All calibration requirements were within acceptance criteria.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one(1) method blank was analyzed. TDS was not detected in TDB009WB. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one(1) LCS was analyzed. TDS was within LCS QC limits in TDB009WL. Refer to LCS summary form for details.

Matrix QC Sample
Sample duplicate was analyzed and RPD was within expected value.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Instrument Calibration	Daily, before and after sample analysis	Analytical balance must be within ± 0.1% of the certified weight	Correct the problem and repeat calibration		
Method Blank	One per preparation batch	Result ≤ ½ LOQ	Re-analyze method blank and all samples processed with the contaminated blank.		
LCS	One LCS per preparation batch	% Recovery: 80% - 120%	Re-analyze the LCS and all associated samples.		
Sample Duplicate	One for every 10 field samples	RPD ≤ 5%	Discuss in the case narrative.		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
20th and 21st Editions			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
TOTAL DISSOLVED SOLIDS
SM 2540C - 1997

SOP: EMAX-2540C Rev. 7
Conc Unit: mg/L
Sample Amount(ml): 20

Analysis date: 10/10/14
Analyzed by: S. Yamauchi

PARAMETER	TDJ004WL	TDJ004WC	TDJ004WX	TDJ004WY	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	TDJ00402	TDJ00403	TDJ00404	TDJ00405							
Total Dissolved Solids	1016	1021	1033	1009	1000	1019	102	10.1	1	80 - 120	PASSED

2540CFA:

ANALYTICAL RUN LOG

Data FileID	Sample ID	Sample Amt(ml)	Dish #	Dish (g)	1stDry Wt+Dish (g)	Date Time	2ndDry Wt+Dish (g)	Date Time	FinalDry Wt+Dish (g)	Date Time	Diff.of Dry Wt. Rdgs (<0.5mg)	Solids (mg)	TDS (mg/L)	Result (mg/L)

Beginning Balance Check			
Std. Wt. (g)	Balance Rdg (g)	Date	Comments
0.1			
5			
100			
Ending Balance Check			
Std. Wt (g)	Balance Rdg (g)	Date	Comments
0.1			
5			
100			
Beginning Balance Check			
Std. Wt (g)	Balance Rdg (g)	Date	Comments
0.1			
5			
100			
Ending Balance Check			
Std. Wt (g)	Balance Rdg (g)	Date	Comments
0.1			
5			
100			

Balance ID: 40706360 Weight ID: 31116

Acceptance Criteria: +/- 0.1% or 0.5 mg whichever is greater

Oven Temp	Reading(°C)	Date	Thermometer ID
Start			
End			
StandardID	Desc.	Conc. (mg/L)	ExpDate
	MB	ND	
	LCS	1000	
	pH strip		

QC Check

LabSampleID	Result	Expected Value	QC Result

Notes: LOQ: 10 mg/L

Micropipette ID:

Analyzedby: _____

SOP

☐ EMAX-160.1 Rev.

☐ EMAX-2540C Rev.

Checked by: _____

Date: _____

SOP REVIEW FORM

EMAX-4500-S2F	Rev.	SULFIDE (TITRIMETRIC, IODINE)
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				Update 6.7
Safety				I have read all SDS of chemicals listed in this SOP.
Instruments, Chemicals & Reagents				
Standards				
Procedures				
- Sample Preparation				
- Instrument Parameters				
- Standardization				
- Analysis				
- Calculations				
- Data Reduction				
- Report Generation				
- Data Review				
- Preventive Maintenance				
Quality Control				
Corrective Action				Update 12.4 to include out of HT for NCR.
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 _____ Date: 10/6/15

FAUNA MADAMBA

STANDARD OPERATING PROCEDURES

SULFIDE (TITRIMETRIC, IODINE)

SOP No.:	EMAX-4500-S2F	Revision No.	3	Effective Date:	01-Aug-13
Prepared By:	Mary Jane Mendoza			Date:	08.01.13
Approved By:	Kenette Pimentel QA Manager			Date:	08-01-13
Approved By:	Caspar Pang Laboratory Director			Date:	08-01-13

Control Number: 4500-S2F-03-

1.0 SCOPE AND APPLICATION

1.1. This method is used to determine total and dissolved sulfides in aqueous samples.

1.2. This method is an adaptation of Standard Method 4500-S^{2-}F-2000 (Iodometric Method), Standard Methods for the Examination of Water and Wastewater, 21st Edition.

1.3. Based on the Methods Update Rule and Title 40 Code of Federal Regulations, Part 136, EPA Method 376.1 was replaced with SM 4500-S^{2-}F-2000. Since EPA Method 376.1 and SM 4500-S^{2-}F-2000 are equivalent methods, where necessary, this SOP is also applicable to EPA Method 376.1.

2.0 SUMMARY OF METHOD

2.1. A known amount of sample is acidified with HCl and oxidized with standard iodine solution to convert sulfides to sulfur. The solution is back titrated with sodium thiosulfate ($Na_2S_2O_3$) standard solution.

2.2. **Interferences**

2.2.1. Reduce sulfur compounds, such as sulfite, thiosulfate and hydrosulfite, which decompose in acid that may yield erratic results. Also, volatile iodine-consuming substances will give high results.

2.2.2. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

2.2.3. If the sample is not preserved with zinc acetate, the analysis must be started immediately. Similarly, the measurement of dissolved sulfides must also be commenced immediately.

2.2.4. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class).

3.0 DETECTION LIMITS

3.1. **Detection Limit (DL) & Limit of Detection (LOD)**

3.1.1. DL and LOD are not applicable for this method.

3.2. **Limit of Quantitation (LOQ)**

3.2.1. Refer to EMAX-QA04 for validation, verification and generation of LOQ.

3.2.2. Established LOQ for this method is 1 mg/L.

4.0 DYNAMIC RANGE

4.1. The maximum amount of titrant used in a pre-determined amount of sample must not exceed 50 ml. All samples analyzed above this range are considered estimate (over-range) and require a smaller amount of sample or higher titrant concentration to properly quantitate.

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Water Samples**

5.1.1. Total Sulfides

5.1.1.1. Samples received in the laboratory are expected to be contained in HDPE bottles, preserved with Zinc Acetate (ZnAc) and Sodium Hydroxide (NaOH), pH > 9, maintained at ≤ 6°C without freezing.

5.1.1.2. Samples must be analyzed within 7 days from sampling time.

5.1.2. Dissolved Sulfides

5.1.2.1. Samples received in the laboratory are expected to be contained in HDPE bottles without preservative.

5.1.2.2. Samples visually free from suspended solids are preserved as in Section 5.1.1.1.

5.1.2.3. Samples with visually present suspended solids are treated as follows:

- Add 0.5 ml (4 drops) 6 N NaOH in 250 ml glass bottle.
- Fill bottle with sample and add 0.5 ml Aluminum Chloride ($AlCl_3$) Solution.
- Rotate for 1 min or longer to flocculate contents.
- Check pH to be 6-9. If pH range is not attained, additional volume of NaOH and $AlCl_3$ can be added.
- Let it settle for about 5-15 mins and decant clear supernatant to a container with ZnAc.
- Check pH to be > 9. Add enough NaOH to raise the pH.

5.1.2.4. Samples must be analyzed within 7 days from sampling time.

5.2. **Soil Samples**

5.2.1. Samples received in the laboratory are expected to be contained in glass jars or brass tubes maintained at ≤ 6°C without freezing.

5.2.2. Soil samples must be extracted within 14 days from date of sampling. Samples must be stored at ≤ 6°C without freezing until analyzed.

5.2.3. Extracts are treated as water samples upon completion of extraction.

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-QC02 Standard Preparation

6.6. EMAX-QC04 Balance Calibration

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 SDS of chemicals listed in this SOP.

7.2. All reagents, standards, and samples are treated 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.

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 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. Titration apparatus ± 0.10 calibration

8.1.2. Magnetic stirrer

8.1.3. Micropipettes

8.1.4. Erlenmeyer flasks, 250 ml

8.1.5. pH strips, 0 - 14

8.2. **Chemicals and Reagents**

8.2.1. All reagents are reagent grade or better.

8.2.2. Reagent water: Type II

8.2.3. Silica Sand

8.2.4. Hydrochloric acid, HCl , 6N(1+1)

8.2.5. Starch or amylose indicator: Commercially available.

8.2.6. Sodium Hydroxide, NaOH, 6N: Dissolve 60 g of NaOH pellets in reagent water and dilute to 250 mL final volume.

8.2.7. Aluminum Chloride ($AlCl_3$) solution: Dissolve 100 g of $AlCl_3$.6H20 in 144 mL reagent water.

8.2.8. Zinc Acetate (ZnAc) solution: Purchase commercially.

9.0 STANDARDS

9.1. Purchase all standards as certified solutions as listed below:

STANDARD	SOURCE	CONCENTRATION
Sulfide Solution	Aqua	1000 mg/L
Iodine Solution	VWR	0.0250 N
$Na_2S_2O_3$ solution	VWR	0.0250 N

9.2. Refer to EMAX-QC02 for proper preparation of analytical standards.

9.3. Store standard solutions at ≤ 6°C without freezing.

9.4. Alternatively, you may prepare the above standards as follows:

9.4.1. Iodine solution @ 0.0250 N. Dissolve 20 to 25 g KI in a little water in a liter volumetric flask and add 3.2 g iodine. Allow to dissolve. Dilute to 1 L.

9.4.2. Sulfide solution @ 100 mg/L. Dissolve 7.49 g with $Na_2S \bullet 9H_2O$ in DI water and dilute to 1 L in a volumetric flask. Standardize against $Na_2S_2O_3$, 0.00564 N.

9.4.3. $Na_2S_2O_3$ solution @ 0.0250 N. Dissolve 3.9535 g of $Na_2S_2O_3$ and dilute to 1 L using DI water.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Aqueous Samples

10.1.1.1. Allow samples to equilibrate to room temperature prior to analysis.

10.1.1.2. Transfer 100 ml sample or appropriate volume in a properly labeled snap seal vial. Let the precipitate settle for 30 minutes or centrifuge.

10.1.1.3. If sample(s) are observed to be clear with no precipitate, then proceed to 10.4, otherwise perform 10.1.1.4 to 10.1.1.7.

10.1.1.4. Decant as much supernatant as possible without loss of precipitate

10.1.1.5. Refill the vial with reagent water back to the original volume of sample.

10.1.1.6. Re-suspend precipitate and proceed to section 10.4.2. Note any unusual matrix observation on the run log.

10.1.1.7. For MB and LCS, use reagent water. Spike the LCS with 0.2 mL of sulfide solution, 1000 mg/L (Section 9.1), and dilute to 100 mL final volume. Mix and proceed to Section 10.4.2.

10.1.2. Soil Samples

10.1.2.1. Determine the appropriate ratio of soil sample and reagent water taking into consideration the project's LOQ.

10.1.2.1.1. Typical soil to reagent water ratio is 10:100.

10.1.2.2. For sludge and sediment samples, check Limit of Quantitation (LOQ) required for the project. Determine the amount of sample needed to be weighed.

10.1.2.3. Weigh a suitable amount of soil to be extracted. Use silica sand for MB and LCS.

10.1.2.4. Add the appropriate amount of reagent water.

10.1.2.5. For LCS and MS, spike with appropriate concentration depending on the project's LOQ.

10.1.2.6. Place the bottle in a shaker at medium speed for 1 hour.

10.1.2.7. Remove the bottle from the shaker and centrifuge the samples for 15 to 30 minutes or allow the particles to settle.

10.1.2.8. Filter the extract through a 0.45 µm membrane if necessary to have a clear sample and add zinc acetate and NaOH. Treat the filtrate as an aqueous sample.

10.2. **Instrument Parameters**

10.2.1. Use burette with a ± 0.10 calibration for titration.

10.3. **Standardization**

10.3.1. Perform triplicate standardization of standard titrant, 0.0250 N sodium thiosulfate against 0.025 N iodine when titrant is prepared in-house.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Method Blank

10.4.1.2. LCS/LCSD

10.4.1.3. Sample Dup

10.4.1.4. Samples (Max of 20 samples per batch)

10.4.2. Analysis of Sample

10.4.2.1. Measure 2.5 ml of standard iodine solution into a 250 ml flask.

10.4.2.2. Add 2.5 ml of 6N HCl.

10.4.2.3. Transfer the 100 ml sample prepared in section 10.1.

10.4.2.4. If the iodine color disappears, add more iodine until the color remains. Record the total amount of standard iodine used.

10.4.2.5. Titrate with the reducing solution or 0.0250 N sodium thiosulfate solution until straw (pale yellow) color is displayed.

10.4.2.6. Add starch solution or amylose indicator and titrate until blue color disappears. Record titrant reading.

10.5. **Calculations**

10.5.1. Calculate for Sample Concentration

10.5.1.1. **For aqueous samples**

$$Sulfide, mg/L = \frac{[(V_I \times C_I) - (V_{ST} \times C_{ST})] \times 16{,}000}{V_S}$$ Eq.-10.5.1.1

where:

V_I – Volume of iodine, mL

C_I – Concentration of iodine, N

V_{ST} – Volume of $Na_2S_2O_3$, mL

C_{ST} – Concentration of $Na_2S_2O_3$, N

V_S – Volume of sample titrated, mL

10.5.1.2. **For soil samples**

$$Sulfide, mg/Kg = \frac{[(V_I \times C_I) - (V_{ST} \times C_{ST})] \times B / C}{\%S}$$ Eq.-10.5.1.2

where:

B – Final extract volume, mL

C – Sample amount, g

$\%S$ – % dry weight of sample = (1-%moisture)/100

10.5.2. Calculate for Percent Recovery

$$\%Recovery = \frac{C_f - C}{C_s} \times 100$$ Eq.-10.5.2

where:

C_f – Concentration found

C – Concentration of the sample (use 0 for LCS)

C_s – Concentration of spike

10.5.3. Calculate for Precision

$$RPD = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)}$$ Eq.-10.5.3

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. Print a copy of the run log.

10.6.2. Make a copy of the sample preparation log (if any).

10.6.3. Highlight the data to be reported.

10.7. **Report Generation**

10.7.1. Generate the method .txt file, sample results and QC Summary using TITRI.exe.

10.7.2. Generate the case narrative using CN2.exe.

10.7.3. Arrange the analysis package in sequence as detailed below.

10.7.3.1. Case Narrative

10.7.3.2. Sample Results

10.7.3.3. LCS/LCSD Summary

10.7.3.4. MS/MSD Summary

10.7.3.5. Sample Duplicate Result Summary

10.7.3.6. Run Log

10.7.3.7. Sample Preparation Log (if any)

10.7.3.8. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01and the PSR.

- Check that MB is compliant to PSR.
- Check LCS/LCD recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
- If any of the above checkpoints is non-compliant, re-analysis is required.
- Check the generated reports against the run log. Check that the analytical data generated indicating positive results are quantitatively correct.
- Review the case narrative and 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. Not applicable.

11.0 QUALITY CONTROL

11.1. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.2. Analytical batch includes ≤ 20 field samples and the following:

- Method Blank (MB)

- Lab Control Sample (LCS)
- Sample Duplicate

11.3. Acceptance criteria are summarized in Appendix 1.

11.4. Method proficiency must be established before the analytical procedure can be used.

11.5. All analysts conducting this analysis must demonstrate capability (IDOC/DOC) as described in EMAX-QA05.

12.0 CORRECTIVE ACTION

12.1. Corrective action for each Quality Control Procedure is summarized in Appendix 1.

12.2. **QC Samples**

12.2.1. Method Blank - If MB is non-compliant consider the following suggestions to analyze and possibly correct the problem:

- Investigate the source of the problem and institute resolution to correct, minimize or eliminate the problem.
- If the analyte found in the 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.

12.2.2. Lab Control Sample(s) - If LCS is non-compliant consider the following suggestions to analyze and possibly correct the problem:

- Check for errors in calculation and concentration of the analyte solution.
- If recovery of LCS is bias-high and associated sample results are non-detect, then results can be reported. If associated samples show positive results or if recovery of LCS is bias-low, then acquire or prepare a new LCS standard and titrant and re-analyze with the associated samples.
- If recovery of LCS is still non-compliant after re-analysis, check the PSR for corrective action. Otherwise, discuss the issue in the case narrative.

12.3. For insufficiency of sample(s) / chemical preservatives, inform the Supervisor immediately for further advice.

12.4. Other anomalies encountered during the analytical process that is not listed in Appendix 1 requires a non-conformance report (NCR). Refer to EMAX-QA08.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of standards and reagents that may go to wastewater drains.

14.0 WASTE MANAGEMENT

14.1. No samples shall dumped on the laboratory sink.

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. 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.

15.1.1.2. **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.

15.1.2. 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.

15.1.3. Safety Data Sheet (SDS) – is where the physical data, toxicology and safety precaution of a certain substance is listed.

15.1.4. Reagent Water – Purified water free from any target analyte or any other substances that may interfere with the analytical process.

15.1.5. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.

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. Lab Control Sample Duplicate (LCSD) – is a replicate of LCS analyzed to monitor precision in the absence of MS/MSD sample.

15.1.8. 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.

15.1.9. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.10. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.11. Matrix – is a component or form of a sample.

15.1.12. 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.

15.1.13. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

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. Standard Methods for the Examination of Water and Wastewater, 21st Edition, Method 4500-S^{2-}F-2000

16.2. Standard Methods for the Examination of Water and Wastewater, 21st Edition, Methods 4500-S^{2-}B-2000 and 4500-S^{2-}C -2000

16.3. Methods for Chemical Analysis of Water and Wastes (EPA/600/4-79/020), EPA Method 376.1, 1978.

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. **Figures**

17.1.1. Figure 1 Analytical Flow Path for Sulfide Determination

17.1.2. Figure 2 Typical Sample Result Summary

17.1.3. Figure 3 Typical LCS/LCSD Summary

17.1.4. Figure 4 Typical Sample Duplicate Result Summary

17.1.5. Figure 5 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. 4500-S2FFA Analytical Run Log

Figure 1: ANALYTICAL FLOW PATH FOR SULFIDE DETERMINATION

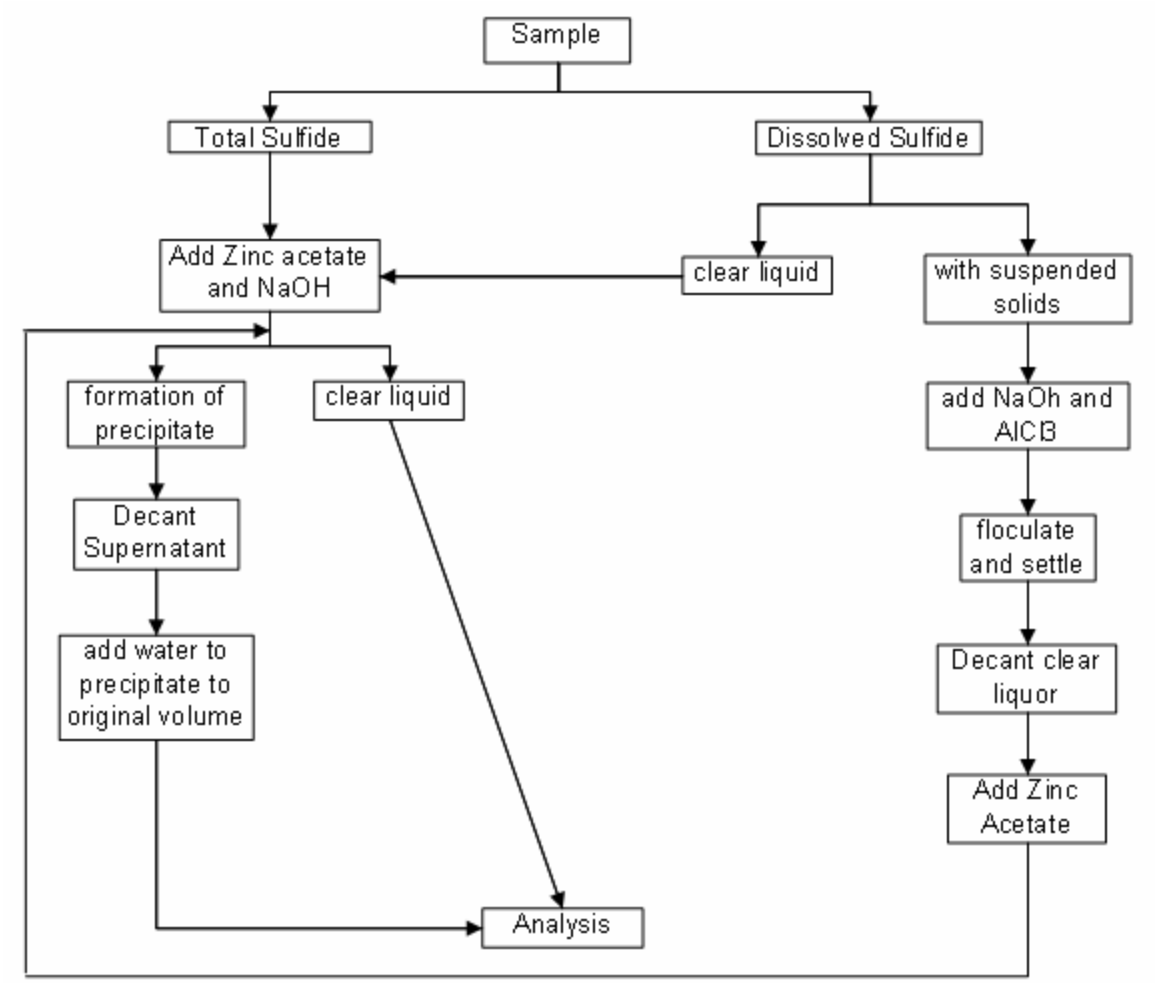


Figure 2: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD 4500-S2F
SULFIDE

Client : XYZ, INC.
Project : CLEAN WATER PROJECT
Batch No. : 13C076

Matrix : WATER
InstrumentID : NA

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	PREP. FACTOR	MOIST (%)	RL (mg/L)	MDL (mg/L)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
MBLK1W	SDC001WB	ND	1	NA	1	1	03/18/1314:05	NA	13SDC001W01	13SDC001	SDC001W	NA	NA
LCS1W	SDC001WL	2.16	1	NA	1	1	03/18/1314:20	NA	13SDC001W02	13SDC001	SDC001W	NA	NA
LCD1W	SDC001WC	2.13	1	NA	1	1	03/18/1314:24	NA	13SDC001W03	13SDC001	SDC001W	NA	NA
IR06-031313-26	C076-02	ND	1	NA	1	1	03/18/1314:29	NA	13SDC001W04	13SDC001	SDC001W	03/13/1311:05	03/14/13
IR06-031313-25	C076-03	ND	1	NA	1	1	03/18/1314:34	NA	13SDC001W05	13SDC001	SDC001W	03/13/1312:10	03/14/13
IR06-031313-SB	C076-04	ND	1	NA	1	1	03/18/1314:38	NA	13SDC001W06	13SDC001	SDC001W	03/13/1313:20	03/14/13
IR06-031313-ER	C076-05	ND	1	NA	1	1	03/18/1314:42	NA	13SDC001W07	13SDC001	SDC001W	03/13/1313:40	03/14/13
IR06-031313-ERDuP	C076-05D	ND	1	NA	1	1	03/18/1314:46	NA	13SDC001W08	13SDC001	SDC001W	03/13/1313:40	03/14/13

Figure 3: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LAB CONTROL SAMPLE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER PROJECT
BATCH NO. : 13C076
METHOD : METHOD 4500-S2F

MATRIX :	WATER		% MOISTURE: NA
DILUTION FACTOR:	1	1	1
SAMPLE ID :	MBLK1W	LCS1W	LCD1W
LAB SAMPLE ID :	SDC001WB	SDC001WL	SDC001WC
LAB FILE ID :	13SDC001W01	13SDC001W02	13SDC001W03
DATE EXTRACTED :	NA	NA	NA
DATE ANALYZED :	03/18/1314:05	03/18/1314:20	03/18/1314:24
PREP BATCH :	SDC001W	SDC001W	SDC001W
CALIBRATION REF:	13SDC001	13SDC001	13SDC001

ACCESSION:

PARAMETER	MB RESULT (mg/L)	SPIKE AMT (mg/L)	BS RESULT (mg/L)	BS REC (%)	SPIKE AMT (mg/L)	BSD RESULT (mg/L)	BSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
SULFIDE	ND	2	2.16	108	2	2.13	107	1	80-120	20

Figure 4: TYPICAL SAMPLE DUPLICATE RESULT SUMMARY

EMAX QUALITY CONTROL DATA
SAMPLE DUPLICATE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER PROJECT
BATCH NO. : 13C076
METHOD : METHOD 4500-S2F

MATRIX :	WATER	
DILUTION FACTOR:	1	1
SAMPLE ID :	IR06-031313-ER	IR06-031313-ERDUP
LAB SAMPLE ID :	C076-05	C076-05D
LAB FILE ID :	13SDC001W07	13SDC001W08
DATE PREPARED :	NA	NA
DATE ANALYZED :	03/18/1314:42	03/18/1314:46
PREP BATCH :	SDC001W	SDC001W
CALIBRATION REF:	13SDC001	13SDC001

ACCESSION:

PARAMETER	PARENT RESULT (mg/L)	DUP RESULT (mg/L)	RPD (%)	MAX RPD (%)
SULFIDE	ND	ND	0	20

Figure 5: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER PROJECT

SDG : 13C076

METHOD 4500-S2F
SULFIDE

A total of four (4) water samples were received on 03/14/13 for Sulfide analysis, Method 4500-S2F in accordance with Standard Methods for the Examination of Water and Wastewater, 21st Edition.

Holding Time
Samples were analyzed within the prescribed holding time.

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 SDC001WL/C were all within QC limits.

Matrix QC Sample
No matrix QC sample was designated for this SDG.

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	2nd Rvw
Method blank	One per preparation batch	Analyzed detected ≤ LOQ	Re-prep and re-analyze method blank and all samples processed with the contaminated blank.		
LCS	One LCS per preparation batch	Within project limit *%Rec. 80-120%	Re-prep and re-analyze the LCS and all associated samples.		
Sample Duplicate	One sample duplicate per every 20 project samples per matrix	Within project limit *RPD ≤ 20%	If LCS is in control and no apparent reason for MD to be outside the QC limits, discuss that MD result is indicative of matrix interference in case narrative.		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
21st Edition			Date:		

* in the absence of project limit default to

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
SULFIDE
SM 4500 S2F

SOP: EMAX- -4500S2F
Conc Unit: mg/L
Sample Amount(ml): 100

Analyzed date: 7/9/2012
Analyzed by: G. Gatbonton

PARAMETER	SDG001WL	SDG001WC	SDG001WX	SDG001WY	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	SDG001-02	SDG001-03	SDG001-04	SDG001-05							
Sulfide	3.92	3.88	3.92	3.96	4.66	3.92	84	0.033	0.8	80 - 120	PASSED

4500-S2FFA:

ANALYTICAL RUN LOG

Prep BatchID	Lab SampleID	Aliquot	Unit	Final Vol (ml)	Vol Titrated (ml)	Iodine (ml)	$Na_2S_2O_3$ (ml)	Date Time	Moisture %	Sulfide (WetBasis)	Sulfide (DryBasis)	Result	Unit	Notes

Standard/ Reagent ID	Description	Concentration	Exp.Date
	Iodine (N)	0.0252	
	LCS Sulfide (mg/L)	8.48	
	LCS Sulfide (mg/Kg)	76.2	
	$Na_2S_2O_3$(N)	0.0245	
	HCl (N)	6	
	Starch		
	Reagent Water		

☐ Standard Purchased as Certified Solution

STANDARDIZATION			
Iodine (ml)	$Na_2S_2O_3$ (ml)	Conc.(N)	DATE
Average Normality of $Na_2S_2O_3$=			

Micropipette ID:

QC Check	Result	Expected Value	Comment

Comment:

☐ EMAX-376.1 Rev. 5 Analyzed By:

☐ EMAX-4500S2F Rev. 2 Checked By:

Date:

SOP REVIEW FORM

EMAX-5310B	Rev. 3	TOTAL ORGANIC CARBON
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 Preparations	✓			
- Instrument Parameters	✓			
- Calibration	✓			
- Analysis	✓			
- Sample Result Evaluation	✓			
- 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 Carolyn Carson Yu Date: 8/3/16

STANDARD OPERATING PROCEDURES

TOTAL ORGANIC CARBON

SOP No.:	EMAX-5310B	Revision No.	3	Effective Date:	29-Apr-13
Prepared By:	Lucita Arzadon			Date:	04-29-13
Approved By:	Kenette Pimentel QA Manager			Date:	04.29.13
Approved By:	Caspar Pang Laboratory Director			Date:	04-29-13

Control Number: 53010B-02

1.0 SCOPE AND APPLICATION

1.1. This method is used to determine total organic carbons (TOC) in an aqueous sample by combustion-infrared technique. This procedure is only applicable to homogenous samples.

1.2. This SOP is an adaptation of Method 5310B-2000, Standard Methods for the Examination of Water and Wastewater, 21st Edition.

2.0 SUMMARY OF METHOD

2.1. An aliquot of acidified sample is injected into the Analyzer, sparged with gas (compressed air or nitrogen) to remove IC content, and heated at 680°C to convert IC to Total Organic Compound.

2.2. **Interference**

2.2.1. Carbonate and bicarbonate carbon represents interference and must be removed or accounted for in the final calculation.

2.2.2. Suspended particles of a liquid sample or extract, is known to alter results.

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 (mg/L)	0.25	0.5	1.0

4.0 DYNAMIC RANGE

4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point. 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 & PRESERVATION

5.1. **Sample Collection**

5.1.1. Samples received in the laboratory are expected to be contained in HDPE or glass bottles and cooled to ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. Samples are expected to be stable for 28 days.

5.3. **Preservation**

5.3.1. Preserve samples at pH <2 with either HCl or H_2SO_4.

5.3.2. Store samples in plastic or glass containers that prevent any possible carbon contamination.

5.3.3. Store samples at ≤ 6°C without freezing.

6.0 ASSOCIATED SOPs

6.1. EMAX-DM1 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 for 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 SDS 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.

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 tap 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

8.1.1. Total Organic Carbon Analyzer Model TOC-V_{CPN} or equivalent

8.1.2. Autosampler – ASI-V

8.1.3. Filtering apparatus and 0.45-µm-pore diam filters, preferably HPLC syringe filters or similar.

8.1.4. Micropipettes – 1 and 5 ml, 200 µl

8.2. Chemicals and Reagents

8.2.1. All reagents are reagent grade or better.

8.2.2. Hydrochloric Acid, 2M HCl

8.2.3. Sulfuric Acid, concentrated H_2SO_4

8.2.4. Reagent Water

9.0 STANDARDS

9.1. Standard Preparation

9.1.1. Refer to EMAX-QC02 for proper preparation of analytical standards.

9.1.2. Oven dry Potassium Hydrogen Phthalate @ 105°C - 120°C for 1- hour.

9.1.3. Oven dry Sodium Hydrogen Carbonate @ 280°C - 290°C for 1- hour.

9.1.4. All standards are cooled and stored in dessicator.

9.1.5. Other concentration levels may be prepared other than specified below as long as it is compliant to required acceptance criteria, and should be stored at ≤ 6°C without freezing.

9.2. Stock Standards

9.2.1. Primary Stock Standards – are purchased commercially as certified standards. These standards are primarily used for calibration.

Standard Name	Source	Concentration (mg/L)
Total Organic Carbon	ERA or equivalent	1,000

9.2.2. Secondary Stock Standards – are purchased commercially from different sources. These standards are used for ICV and LCS.

Standard Name	Source	Concentration (mg/L)
Potassium Phthalate, crystal	Mallinckrodt or equivalent	Assay

9.2.3. Other Standards – are purchased commercially and these standards are used to determine the removal of carbonate and bicarbonate by purging.

Standard Name	Source	Concentration (mg/L)
Sodium Bicarbonate	BDH or equivalent	Assay
Sodium Carbonate	Mallinckrodt or equivalent	Assay

9.3. **Intermediate Standards**

9.3.1. Secondary Intermediate Standards – are prepared using the secondary stock standards as follows:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
Potassium Phthalate	Assay	0.2128 g	1,000 mg/L	Reagent Water	100 ml

9.3.2. Carbonate/Bicarbonate Intermediate Standards – is prepared as follows:

Standard Name	Concentration	Amount	Final Conc.	Solvent	Final Volume
Sodium Carbonate	Assay	0.4418 g	1,000 mg/L	Reagent Water	100 ml
Sodium Bicarbonate	Assay	0.3512			

9.4. **ICAL Standards**

9.4.1. A minimum of 5 point calibration standards are prepared using a clean 100 ml volumetric flask, in reagent water.

ICAL Pt.	Primary Stock Standard (1,000 mg/L) Aliquot (µl)	Final Volume (ml)	Concentration (mg/L)
S0	0	100	0
S1	0.1	100	1
S2	0.5	100	5
S3	1.0	100	10
S4	4.0	100	40
S5	8.0	100	80

9.5. **Check Standards**

9.5.1. Initial Calibration Verification (ICV)

9.5.1.1.1. Prepare ICV standard by diluting 2.5 ml of secondary intermediate standard (1000 mg/L) solution to 100 ml reagent water. The expected value of this is 25 mg/L.

9.5.2. Continuing Calibration Verification (CCV)

9.5.2.1.1. Prepare CCV standard by diluting 2.5 ml of primary stock standard solution to 100 ml reagent water. The final concentration of the check standard shall be 25 mg/L.

9.5.3. Carbonate/Bicarbonate Standard

9.5.3.1. Prepare Carbonate/Bicarbonate standard by diluting 0.1 ml of Carbonate/Bicarbonate intermediate standard (1,000 mg/L) to 100 ml of reagent water. The expected value of this is 1 mg/L.

10.0 PROCEDURES

10.1. Sample Preparations

10.1.1. When Dissolved Organic Carbon (DOC) is to be determined, filter the sample prior to injection. Otherwise, aqueous sample is directly injected into the instrument.

10.2. Instrument Parameters

10.2.1. TOC-V_{CPN}

Catalyst Type	TOC High Sensitivity Catalyst
Furnace Temperature	680°C
Gas Flow Rate	150 ml/min
Tubing Diameter	0.5 min.
NDR Cell Length	Long
Range	X3
Replicate Injection	2

10.2.2. Autosampler : ASI-V

Tray Type	24 ml vial
Needle	Sample needle
Needle Wash/Flow Line Wash	2
Rack	0 – 93

10.3. Calibration

10.3.1. Analyze the ICAL standards. Refer to Section 9.4.

10.3.2. Generate the calibration curve after the multi-level ICAL analysis is completed.

10.3.3. Analyze ICV to verify the ICAL.

10.3.4. Refer to Appendix 1 for acceptance criteria.

10.4. Analysis

10.4.1. Place ICAL standards, check standards and sample in designated Auto Sampler positions.

10.4.2. Set up calibration(*.cal)\method(*.met)\template(*.tpl) parameters, refer to Shimadzu TOC-VCPN Sections 4.3 to 4.4.

10.4.2.1. If desired calibration\method\template parameter already exist, in C:\Program Files\Shimadzu Corporation\TOC3201, save and rename the templates in their respective folders, such that, 80_9060.cal(4x injections) and/or 80_415.cal(2x injections) as TCI001.cal.

10.4.2.2. Make sure the templates fulfill all project requirements and generate a sequence by opening a new file to include, Calibration curve, Control Calibration Checks and Auto-Generate to program sample positions.

10.4.3. Check background monitor in the instrument menu before starting the analysis. All window should display green check marks.

10.4.4. Set quantitation to average duplicate determination. Refer to NPOC/TOC Criteria Quantitation of TOC results outlined in TOC-V manual Section 5.1.3 and 5.1.5.

10.4.5. Sparge the acidified sample (pH = 2~3) with zero grade air and/or high purity nitrogen gas, to eliminate the IC Component. Measure the TC remaining in the sample and determine the Total Organic Carbon (NPOC).

10.4.6. The ICAL, check standards and samples must be injected in duplication.

10.4.7. Analytical Sequence

10.4.7.1. ICAL – initial calibration

10.4.7.2. ICV – initial calibration verification

10.4.7.3. ICB – initial calibration blank

10.4.7.4. MB - method blank

10.4.7.5. LCS – lab control sample

10.4.7.6. LCSD – lab control sample duplicate (in the absence of MS)

10.4.7.7. Samples - maximum of 10 field samples

10.4.7.8. Duplicate – sample duplicate

10.4.7.9. MS – matrix spike

10.4.7.10. CCV – continuing calibration check

10.4.7.11. CCB – continuing calibration blank

10.4.7.12. Samples - maximum of 10 field samples

10.4.7.13. Duplicate – sample duplicate

10.4.7.14. MS – matrix spike

10.4.7.15. ECC – ending calibration check

10.4.7.16. ECB – ending calibration blank

10.5. **Sample Result Evaluation**

10.5.1. Evaluate QC results as soon as the data is available.

- ✓ Check CCVs that they are within the acceptance criteria.

- ✓ Check that the batch QC samples met the acceptance criteria.
- ✓ 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, check the specified corrective action in Appendix 1 to determine if re-analysis is necessary. If re-analysis results are the same as the initial result, consult the Supervisor for further action.

10.6. **Calculations**

10.6.1. Calculate for Standard Deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}} \qquad \text{Eq.-10.6.1}$$

where:

X_i – Result at the ith measurement

$\bar{x}$ – Mean

N – Number of measurements

10.6.2. Calculate for % Relative Standard Deviation (%RSD)

$$\%RSD = \left[\frac{SD}{ACF}\right] 100 \qquad \text{Eq.-10.6.2}$$

where:

SD – Standard deviation

ACF – Average Calibration Factor

10.6.3. Calculate for Percent Recovery

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.6.3}$$

where:

C_f – Concentration found

C – Concentration of the sample (use 0 for LCS)

C_s – Concentration of spike

10.6.4. Calculate for Relative Percent Difference (RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} x100 \qquad \text{Eq.-10.6.4}$$

where:

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 analytical run log.

10.7.2. Print a copy of the raw data and the QC report.

10.7.3. Highlight the data to be reported.

10.7.4. Collate the reportable data separating the QC results from the sample results.

10.7.5. Keep all other data generated with the analytical folder marked with "For record only".

10.8. **Report Generation**

10.8.1. Generate the method.txt file using WDB5N.exe.

10.8.2. Generate sample results using MSRB6.exe.

10.8.3. Generate the QC Summary using QCIC.exe.

10.8.4. Generate the sample duplicate result using CQ1N.exe.

10.8.5. Generate the Case Narrative using CN2.exe.

10.8.6. Arrange the analysis package in sequence as detailed below.

10.8.6.1. Case Narrative

10.8.6.2. Sample Results

10.8.6.3. LCS/LCD Summary

10.8.6.4. MS/MSD Summary

10.8.6.5. Sample Duplicate Result

10.8.6.6. Calibration Summary

10.8.6.7. Analytical Run Log

10.8.6.8. Non-Conformance Report (If any)

10.9. **Data Review**

10.9.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.9.2. Review the generated reports for calculation accuracy and that dilution factors are properly applied.

10.9.3. Review the attached logs that they are properly filled.

10.9.4. Check the generated reports against the raw data. Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.

10.9.5. 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.

10.9.6. Submit the analytical folder for secondary review.

10.10. **Preventive Maintenance**

10.10.1. Perform daily instrument check prior to sample analysis. Refer to Form 5310BFM – Instrument Maintenance Log.

Maintenance Activity	Description	Frequency
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis
System Cleaning	Remove dust from fans and vent covers	Every 6 months
Parts Maintenance	Replace worn out parts	Every 6 months
Lubrication	Lubricate mechanical parts	Every 6 months
Flow and Waste lines	Inspect flow and waste lines and replace	Every 6 moths or as necessary
Combustion Tube	Change combustion tube	Every 6 months or as necessary
Absorber	Change CO2 absorber	Every 6 months or as necessary
Scrubber	Change halogen scrubber	Every 6 months or as necessary
Humidifier	Change humidifier water	Every 6 months
Inspection	Perform general inspection of the complete system	Once a year

10.10.2. Maintain an inventory of instrument parts and supplies for routinely maintenance.

10.10.3. For troubleshooting refer to the TOC analyzer operations manual or go to http://www.ssi.shimadzu.com/toc_virtualadvisor/ for virtual troubleshooting assistance.

11.0 QUALITY CONTROL

11.1. Acceptance criteria for each quality control procedure, is summarized in Appendix 1.

11.2. **Analytical Batch QC**

11.2.1. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06 for details.

11.2.2. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2.3. All analytical standards are prepared fresh on the day the analysis is done. Follow the procedures for all analytical standard preparations as described in EMAX-QC02 and document in the analytical standard preparation log.

11.3. **Preparation Batch QC**

11.3.1. A preparative batch includes a MB, LCS, and a maximum of 20 field original samples unless otherwise specified by the project.

11.3.2. MS/MSD is analyzed every 10 samples unless otherwise specified by the project.

11.3.3. In the absence of MS/MSD, a LCS duplicate (LCD) is prepared and analyzed.

11.3.4. Properly treat all lab wares used in the sample preparation as specified in EMAX-QC07.

11.4. **Method QC**

11.4.1. A valid DL and LOD must exist prior to sample analysis. Refer to EMAX-QA04 for details.

11.4.2. 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 action for each quality control procedure is summarized in Appendix 1.

12.2. **Calibration**

12.2.1. When ICAL is not compliant, consider the following suggestions to correct the problem:

12.2.1.1. If the correlation coefficient (r) is out of acceptance criteria, review the results and identify presence of an outlier.

- If one of the standards 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 and analyze a lower concentration.
- If the lowest point returns a bias low response or the peaks are not distinct and sharp, drop the point and analyze a higher concentration.
- Otherwise, optimize the instrument and repeat the ICAL.

12.2.2. When ICV or CCV is not compliant, consider the following suggestions to correct the problem:

- Rule out standard degradation. Prepare a fresh standard and re-analyze.
- If response is low check for leak or if gas pressure is low.
- If response is high, run a blank to rule out contamination. If contamination exists, determine the source of contamination and apply necessary measures to correct the problem. Re-analyze all associated samples that had positive detections.
- Otherwise, inform the supervisor for further instruction.

12.3. **Preparation Batch QC**

12.3.1. For insufficient amount of sample(s), inform the supervisor immediately for further action.

12.3.2. Method Blank (MB) - When MB is not compliant, consider the following suggestions to correct the problem:

- Rule-out instrument contamination by analyzing instrument blank. In the event that instrument contamination is observed perform the following:
  - replace CO_2 absorber
  - replace the Halogen scrubber
  - clean the humidifier tubing
  - replace the content of the dilution bottle with a freshly harvested reagent water. Purge the wash line.
  - re-analyze instrument blank
- If problem persist, perform the decontamination procedure as described in the operations manual or go to http://www.ssi.shimadzu.com/toc_virtualadvisor/.
- If no apparent instrument contamination is observed, re-analyze another reagent blank freshly harvested from the reagent water purifier. If problem persist, inform the Supervisor.
- Re-analyze all samples with positive results. Where re-analysis is not possible, flag the data as described by the PSR and discuss in the case narrative.

12.3.3. Lab Control Sample (LCS) - When LCS in is not compliant, consider the following suggestions to correct the problem:

- Rule out standard degradation. Prepare a fresh standard and re-analyze.
- If recovery is low check for leak or if gas pressure is low. Apply necessary measures to correct the problem and re-analyze all associated samples. Where re-analysis is not possible, flag the data as described by the PSR and discuss in the case narrative.
- If response is high, run a blank to rule out contamination. If contamination exists, determine the source of contamination and apply necessary measures to correct the problem. Re-analyze all associated samples positive results.
- Otherwise, inform the supervisor for further instruction.

12.3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - When MS/MSD in is not compliant, consider the following suggestions to correct the problem:

- Rule out procedural error, e.g., sample was properly spiked, calculations are correct, etc. If procedural error is apparent, re-analyze MS/MSD.
- If matrix interference is suspected, discuss it in the case narrative.

12.4. 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.

12.4.1. 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 the wastewater drains.

13.2. Prepare all standards in fume hoods to prevent air pollution in the work area.

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. 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.

15.1.1.2. **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.

15.1.2. 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%.

15.1.3. 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%.

15.1.4. 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.

15.1.5. 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.

15.1.6. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.7. Calibration Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.8. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

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. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.14. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.1.15. Matrix – is a component or form of a sample.

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. 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 Total Organic Carbon analysis. In instances where there is a project or program QAPP, the requirement 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. Standard Methods for Examination of Water and Wastewater, 21st Edition

16.2. Title 40 Code of Federal Regulations, Part – 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, latest edition

16.3. Instructional Manual TOC-VCSH/CSN & TOC-Control V Software TOC-VCPH/CPN

16.4. EMAX Quality Systems Manual, as updated

17.0 APPENDICES

17.1. **Figures**

17.1.1. Figure 1 Typical Raw Data

17.1.2. Figure 2 Typical Sample Result Summary

17.1.3. Figure 3 Typical LCS/LCSD Report Summary

17.1.4. Figure 4 Typical MS/MSD Summary

17.1.5. Figure 5 Typical Sample Duplicate Result Summary

17.1.6. 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. Form 5310BFA Analytical Run Log

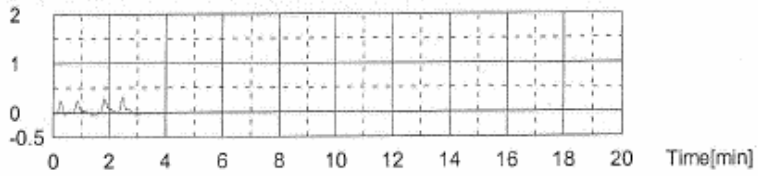
17.3.2. Form 5310BFM Instrument Maintenance Log

Figure 1: TYPICAL RAW DATA

ANDY 11/18/10 11:48:33 AM TCK005.132

No.	Area	Conc.	Inj. Vol.	Aut. Dil.	Ex.	Cal. Curve	Date / Time
1	0.2115	0.1004mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:41:34 AM
2	0.2859	0.1357mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:42:44 AM
3	0.2736	0.1299mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:43:34 AM
4	0.3948	0.1874mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:44:41 AM

Mean Area 0.2914
Mean Conc. 0.1383mg/L



Sample

Sample Name: 10K113-01
Sample ID: TCK005-51
Origin: TCK005.cal
Chk. Result

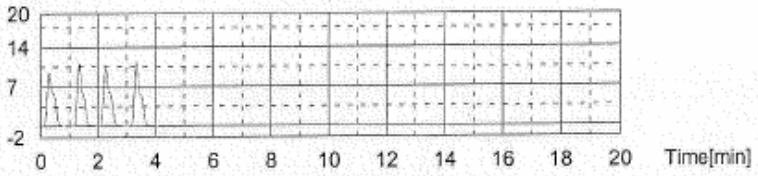
Type	Anal.	Dil.	Result
Unknown	NPOC	1.000	NPOC:7.343 mg/L

1. Det

Anal.: NPOC

No.	Area	Conc.	Inj. Vol.	Aut. Dil.	Ex.	Cal. Curve	Date / Time
1	15.27	7.248mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:52:14 AM
2	15.24	7.234mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:53:22 AM
3	15.62	7.414mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:54:38 AM
4	15.75	7.476mg/L	50uL	1		TCK005.2010_11_17_14_20_55.cal	11/18/10 02:55:53 AM

Mean Area 15.47
Mean Conc. 7.343mg/L



Sample

Sample Name: 10K113-02
Sample ID: TCK005-52
Origin: TCK005.cal
Chk. Result

Type	Anal.	Dil.	Result
Unknown	NPOC	1.000	NPOC:21.40 mg/L

1. Det

Anal.: NPOC

Figure 2: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD SM5310B
TOC

Client : XYZ, INC.
Project : CLEAN WATER PROJECT
Batch No. : 13C501

Matrix : WATER
Instrument ID : I62

SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/L)	DLF	MOIST	RL (mg/L)	MDL (mg/L)	Analysis DATETIME	Extraction DATETIME	LFID	CAL REF	PREP BATCH	Collection DATETIME	Received DATETIME
MBLK1W	TCC003WB	ND	1	NA	1.00	0.500	03/14/1316:01	NA	TCC003-05	TCC003-02	TCC003W	NA	NA
LCS1W	TCC003WL	24.8	1	NA	1.00	0.500	03/14/1316:13	NA	TCC003-06	TCC003-02	TCC003W	NA	NA
LCD1W	TCC003WC	24.4	1	NA	1.00	0.500	03/14/1316:26	NA	TCC003-07	TCC003-02	TCC003W	NA	NA
BARGE	C501-01	ND	1	NA	1.00	0.500	03/14/1317:54	NA	TCC003-15	TCC003-13	TCC003W	03/12/1307:53	03/12/13
SWEETWATER	C501-02	ND	1	NA	1.00	0.500	03/14/1318:05	NA	TCC003-16	TCC003-13	TCC003W	03/12/1308:14	03/12/13
SAN DIEGO	C501-03	2.95	1	NA	1.00	0.500	03/14/1318:16	NA	TCC003-17	TCC003-13	TCC003W	03/12/1308:48	03/12/13
SAN DIEGODUP	C501-03D	2.96	1	NA	1.00	0.500	03/14/1318:27	NA	TCC003-18	TCC003-13	TCC003W	03/12/1308:48	03/12/13
SAN DIEGOMS	C501-03M	28.2	1	NA	1.00	0.500	03/14/1318:39	NA	TCC003-19	TCC003-13	TCC003W	03/12/1308:48	03/12/13
SAN DIEGOMSD	C501-03S	28.3	1	NA	1.00	0.500	03/14/1318:52	NA	TCC003-20	TCC003-13	TCC003W	03/12/1308:48	03/12/13

Figure 3: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 13C501
METHOD: METHOD SM5310B

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	TCC003WB	TCC003WL	TCC003WC		
LAB FILE ID:	TCC003-05	TCC003-06	TCC003-07		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	NA
DATE ANALYZED:	03/14/1316:01	03/14/1316:13	03/14/1316:26	DATE RECEIVED:	NA
PREP. BATCH:	TCC003W	TCC003W	TCC003W		
CALIB. REF:	TCC003-02	TCC003-02	TCC003-02		

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 (%)
TOC	ND	25	24.8	99	25	24.4	97	2	80-120	20

Figure 4: **TYPCAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 13C501
METHOD: METHOD SM5310B

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	SAN DIEGO				
LAB SAMP ID:	C501-03	C501-03M	C501-03S		
LAB FILE ID:	TCC003-17	TCC003-19	TCC003-20		
DATE EXTRACTED:	NA	NA	NA	DATE COLLECTED:	03/12/13 08:48
DATE ANALYZED:	03/14/1318:16	03/14/1318:39	03/14/1318:52	DATE RECEIVED:	03/12/13
PREP. BATCH:	TCC003W	TCC003W	TCC003W		
CALIB. REF:	TCC003-13	TCC003-13	TCC003-13		

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 (%)
TOC	2.95	25	28.2	101	25	28.3	101	0	80-120	20

Figure 5: TYPICAL SAMPLE DUPLICATE RESULT SUMMARY

EMAX QUALITY CONTROL DATA
DUPLICATE SAMPLE ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
BATCH NO.: 13C501
METHOD: METHOD SM5310B

MATRIX:	WATER		% MOISTURE:	NA
DILUTION FACTOR:	1	1		
SAMPLE ID:	SAN DIEGO			
EMAX SAMP ID:	C501-03	C501-03D		
LAB FILE ID:	TCC003-17	TCC003-18		
DATE EXTRACTED:	NA	NA	DATE COLLECTED:	03/12/13 08:48
DATE ANALYZED:	03/14/1318:16	03/14/1318:27	DATE RECEIVED:	03/12/13
PREP. BATCH:	TCC003W	TCC003W		
CALIB. REF:	TCC003-13	TCC003-13		

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	DUPL RSLT (mg/L)	RPD RSLT %	QC LIMIT (%)
TOC	2.95	2.96	0	20

Figure 6: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER PROJECT

SDG : 13C501

METHOD SM5310B
TOC

A total of three (3) water samples were received on 03/12/13 for Total Organic Carbon analysis, Method SM5310B in accordance with Standard Methods for the Examination of Water and Wastewater, 21st Edition.

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 TCC003WL/C were all within QC limits.

Matrix QC Sample
Matrix QC sample was analyzed at the frequency prescribed by the project.
Percent recoveries for C501-03SM/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 1: SUMMARY OF QUALITY CONTROL PROCEDURES

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
ICAL - Minimum of 5-point calibration	Prior to sample analysis	Correlation coefficient (r) ≥ 0.995	Correct the problem then repeat initial calibration		
Initial Calibration Verification (ICV)	Every after ICAL	%Recovery: ± 10% of expected value	Re-analyze ICV to rule out bad injection and/or standard degradation. Otherwise, repeat calibration		
Calibration verification	Every 15 samples and at the end of analysis sequence	%Recovery: ± 10% of expected value	Re-analyze CCV to rule out bad injection and/or standard degradation. Otherwise, repeat calibration and re-analyze all samples since last successful calibration verification.		
Replicate Injection	Every injection	RPD ≤ 10%	Re-analyze		
Method Blank	One per preparation batch	No analytes detected > ½ LOQ	If instrument blank is clean, re-analyze MB and all samples with positive results.		
LCS	One per preparation batch	%Rec: 80-120%	Re-prep and re-analyze the LCS and all associated samples		
Dup/MS	Refer to PSR otherwise, one set every 10 samples	%Rec: 80-120% %D ≤ 20%	If no procedural error is observed, and bracketing CCVs are in control, matrix interference is suspected. Discuss finding in case narrative. Otherwise, re-prep and re-analyze MS/MS and parent sample.		
Comments: Refer to PSR for flagging criteria.			Reviewed By:		
21st Edition			Date:		

Appendix 2: DEMONSTRATION OF CAPABILITY

DEMONSTRATION OF CAPABILITY
TOTAL ORGANIC CARBON
SM 5310B

SOP: EMAX-415.1 / -5310B / -9060
Conc Unit: mg/L
Sample Amount(ml): 1

Analyzed date: 2/24/2011
Analyzed by: Kyaw Linn

PARAMETER	TCB007-06	TCB007-07	TCB007-08	TCB007-09	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	TCB010WL	TCB010WC	TCB011WL	TCB011WC							
TOC	25.45	25.34	25.37	25.27	25	25.36	101	0.075	0.3	80 - 120	PASSED

5310BFA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC

ANALYSIS RUN LOG
for
TOC

Page 1

	Date	Time
Start		
End		

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #:	A62-025
Instrument No.:	62
Pipette ID:	039380025
	SW3-01-33
	SI1A-01-4
Balance ID:	40706360
Analytical Sequence:	
Method File:	
Analytical Batch:	

SOP #	Rev. #
☐ EMAX-415.1	
☐ EMAX-5310B	
☐ EMAX-9060	
☐ EMAX-	

STANDARDS ID	CONC (mg/L)
S0	
S1	
S2	
S3	
S4	
S5	
S6	
ICV/LCS	
CCV	

ELECTRONIC DATA ARCHIVAL	
Location	Date
☐ TOC	
☐	

Reagent Water ID #:

pH Strips Lot #:

2 M HCl

Analyzed By:

Date:

5310BFM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No. IC Month: Year: 201_ Book # MIC-08-

Date							
Air Tank							
Air Pressure (150ml/min)							
Humidifier Water Level							
Pure Water Trap Level							
Rinse Bottle							
Dilution Water Bottle							
External Acid Bottle							
Halogen Scrubber							
CO_2 Absorber							
Background Monitor:							
1) Furnace: 680°C							
2) NDIR: Baseline Position							
Baseline Fluctuation							
Baseline Noise							
3) Dehumidifier: 1,000 reading							
Printer Paper							
Sampler Needle							
Computer Clock							
Analyst Initial							
Trouble Shooting							
Problem(s) Encountered							
Return To Specs (Analyst Initials)							

Code	Problems Encountered	Code	Corrective Action

EMAX
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

SOP QA REVIEW FORM

EMAX-6010	Rev. 7	INDUCTIVE COUPLED PLASMA EMISSION SPECTROMETRIC METHOD FOR TRACE METAL ANALYSES
SOP No.	Revision Number	Title

SECTION	✓	COMMENTS
Scope & Application	✓	
Summary of Method	✓	
Detection Limits	✓	
Dynamic Range	✓	
Sample Holding Time & Preservation	✓	
Associated SOPs	✓	Add EMAP-2006.
Safety	✓	
Instruments, Chemicals & Reagents	✓	
Standards	✓	
Procedures	✓	
- Sample Preparation	✓	
- Instrument Parameters	✓	
- Calibration	✓	
- Analysis	✓	
- Calculations	✓	
- Report Generation	✓	
- Data Review	✓	
- Preventive Maintenance	✓	
Quality Control	✓	
Corrective Action	✓	
Pollution Prevention	✓	
Waste Management	✓	
Supplementary Notes	✓	
References	✓	
Appendices	✓	

No revision necessary.

Reviewed By FAIZNA MADAMBA Date: 5/18/16

STANDARD OPERATING PROCEDURE

INDUCTIVE COUPLED PLASMA EMISSION SPECTROMETRIC METHOD FOR TRACE METAL ANALYSES

SOP No.:	EMAX-6010	Revision No.	7	Effective Date:	27-Oct-10
Prepared By:	Mary Jane Mendoza			Date:	10.27.10
Approved By:	Kenette Pimentel QA Manager			Date:	10.27.10
Approved By:	Caspar Pang Laboratory Director			Date:	10-27-10
		Control Number:	**6010-07-**		

1.0 SCOPE AND APPLICATION

1.1. This procedure is applicable for trace metal quantitative analyses of drinking water, wastewater, groundwater, aqueous, extract, soil, sludge, and sediment samples using the Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) method. All matrices require proper sample preparation prior to analysis.

1.2. The elements and their corresponding wavelengths are listed in Table 1.

1.3. This SOP is an adaptation of the SW846 Method 6010B.

2.0 SUMMARY OF METHOD

2.1. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) is employed to determine individual elements through their respective atomic or energy emission at different wavelengths. Samples are nebulized and the generated aerosol is carried through the center of the plasma torch, where the metals atoms are excited into high electronic states. Atomic and ionic line emission spectra characteristics of the particular metals are produced when the electron decays back to the lower energy levels. The emission is dispersed by a grating spectrometer and the intensities of the specific lines are monitored simultaneously by photomultiplier tubes. The photocurrent produced by the photomultiplier tube will increase in direct proportion to the concentration of the element in the sample within the linear range of a specific emission line. The photocurrent is processed by a computer system and related to concentration through a calibration procedure. The calibration procedure is made on the assumption that the instrumental response is linear with concentration within the dynamic range.

2.2. Calibration is performed by standardizing the instrument with a blank that is matrix matched to the digestate and two point standard. The calibration curve is verified by another standard from a different source before any sample is analyzed. The intensity of emission of each element is measured and compared to a calibration curve for each element concentration versus intensity.

2.3. **Interference**

2.3.1. **Physical Interference.** Contamination might be introduced by the memory effect of metals during previous analysis (carry over) adhering to the walls of the pumping system and the nebulization chamber. Adequate rinsing of at least 125 seconds using matrix acid blank (refer to Section 9.2) shall be performed between sample analyses to minimize system contamination.

2.3.2. **Chemical Interference.** Matrix matching between digestates and all standard and samples dilution is used in sample analysis to eliminate/minimize chemical interference.

2.3.3. **Spectral Interference.** Both off-peak (background) and on-peak (inter-element correction coefficients) interference corrections are applied for all affected elements to take care of spectral interference. Refer to Section 10.3.4.

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 7.

4.0 DYNAMIC RANGE

4.1. The upper limit of the linear dynamic range (LDR) is established for each analyte by determining the signal responses from three to five diff. conc. the range, one of which is rear the upper limit of the range.

4.2. Verify the upper limit of the LDR every six months or when there is a significant change in the instrument signal.

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Water Samples**

5.1.1. Water samples are collected in polyethylene or glass bottles.

5.1.2. For total recoverable metal analysis, the sample is preserved at the time of collection to pH < 2 with nitric acid.

5.1.3. For dissolved metal analysis, the sample is filtered through a 0.45 µm filter at the time of collection and preserved to pH < 2. Samples requested to be properly preserved in the lab shall observe at least 24 hours from the time preservative is added before sample digestion.

5.1.4. Samples shall be stored in the same condition as received unless specified in the project requirement.

5.2. **Soil Samples**

5.2.1. Soil samples are collected in glass jars or brass tubes.

5.2.2. Soil samples are stored at ≤ 6°C until analysis.

5.3. **Holding Time**

5.3.1. All samples must be analyzed within 180 days from the collection date.

6.0 ASSOCIATED SOPs

6.1. EMAX-QC01 - Quality Control for Chemicals

6.2. EMAX-QC02 - Analytical Standard Preparation

6.3. EMAX-QA04 - IDL/MDL/LOQ

6.4. EMAX-QC07 - Glassware Cleaning

6.5. EMAX-QA08 - Corrective Action

6.6. EMAX-SM04 - Analytical and QC Labeling

6.7. EMAX-3005 - Acid Digestion, Total Recoverable or Dissolved Metals

6.8. EMAX-3010 - Acid Digestion, Total Metals (Aqueous)

6.9. EMAX-3050 - Acid Digestion, Total Metals (Solids)

7.0 SAFETY

7.1. Read all MSDS of chemicals listed in this SOP.

7.2. During operation or maintenance of the instrument take the following precautions:

- Close the instruments hoods and panels prior to operation.
- Check the exhaust system for a positive extraction at the exhaust duct.
- Handle the solvents correctly.
- 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 Thermo iCAP 6000.

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. Place all wastes generated during analytical process in the waste containers. Endorse these wastes to waste disposal section for proper disposal.

7.5. Place all wastes generated during digestion, i.e., filter papers, paper towels, etc., and/or any solid material contaminated with acid, in the wastes containers under the fume hoods. Label these wastes acidic wastes and give to the waste management unit for proper disposal. DO NOT DISPOSE ACIDIC WASTE IN THE TRASH CAN.

7.6. Label all acidic rinsate (pH$\leq$2) acidic water and give to the waste management unit for proper disposal. DO NOT DISPOSE ACID WASTE IN THE SINK.

7.7. 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: ID8 – Thermo Electron iCAP 6500 Duo

8.1.2. Autosampler: CETAC ASX-520

8.1.3. Computer: IBM Compatible

8.1.4. RF Generator: Thermo Electron solid state

8.1.5. Data Acquisition: Thermo iTEVA version 2.3.0.56

8.1.6. Autosampler rack(s): 12x5 positions

8.1.7. Culture tubes: 17x100mm, polypropylene

8.1.8. Volumetric Flask: 100, 250, 500, 1000 ml

8.1.9. Micropipettes: 100, 1000, 5000, 10000 µl

8.1.10. Pipette Tips: 200, 1000, 5000, 10000 µl

8.1.11. Polyethylene bottles: 125, 250, 500, 1000 ml

8.1.12. Liquid argon

8.1.13. Liquid nitrogen

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

9.0 STANDARDS

9.1. **Stock Standard**

9.1.1. The stock standards are purchased as certified single element solutions in 1000 mg/L and 10,000 mg/L or as custom-made certified mixed stock solutions in various concentrations.

9.1.2. Purchase custom-made certified mixed stock standard solutions, preferably as suggested in Table 2. Other concentrations or mixes may be purchased where analyte stability is established.

9.1.3. Detailed procedure of standard preparation and labeling is described in EMAX-QC02 and EMAX-SM04, respectively.

9.2. **Matrix Acid Blank (S0)**

9.2.1. The matrix acid blank is used for dilutions of the standards and, digestates, if necessary.

9.2.2. Prepare this solution by mixing 6% by volume nitric acid and 10% by volume hydrochloric acid in reagent water. Transfer into a clean HDPE bottle and identify the solution as S_0.

9.3. **Initial Calibration Standard (ICAL)**

9.3.1. Prepare the ICAL standards to a final volume of 1 liter using S_0 for dilution as suggested in Table 3.

9.4. **Initial Calibration Verification (ICV)**

9.4.1. From a secondary source standard, prepare ICV standard to a final volume of 100 ml using S_0 for dilution, as suggested in Table 4.

9.5. **Continuing Calibration Verification (CCV)**

9.5.1. Using the ICAL standard source, prepare CCV standard to a final volume of 1 liter using S_0 for dilution, as suggested in Table 5.

9.6. **Interference Check Standard, Primary Interferents A (ICSA) and Analytes B (ICSAB)**

9.6.1. Prepare ICSA and ICSAB Standards to a final volume of 1 liter using S_0 for dilution, as suggested in Table 6.

9.7. **Application of Analytical Standards**

9.7.1. All standard concentrations described in Section 9 are typical concentrations. Other standard concentrations may be used as necessary in consideration of project requirements and/or for conformance purposes. Where changes are made, apply the same Method QC requirements and/or project requirements to validate acceptability.

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. For aqueous samples to be analyzed for total recoverable metals and/or dissolved metals, refer to EMAX-3005.

10.1.2. For aqueous samples to be analyzed for total and dissolved metals, refer to EMAX-3010.

10.1.3. For solid samples to be analyzed for total metals, refer to EMAX-3050.

10.2. **Instrument Parameters**

10.2.1. Thermo ICAP 6500 Duo (ID8)

- RF Power: 1150 W
- Pump Rate : 45 RPM
- Auxillary Gas Flow : 0.5 L/min
- Nebulizer Gas Flow : 0.55 L/min
- Coolant Gas Flow : 14
- Purge Gas Flow : Normal
- No. Lines: 33
- Analysis Time: 5 minutes
- No. of Replicates: 3
- Internal Standard: Scandium, 5 mg/L

10.3. **Calibration**

10.3.1. **Instrument Set-Up**

10.3.1.1. Set up ID8 to proper operating parameters. Refer to Section 10.2.1.

10.3.1.2. Ignite the plasma and allow the instrument to become thermally stable at least 30 mins.

10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.2. **Initial Calibration**

10.3.2.1. Set the system to flush with the matrix acid blank between each analysis run for a minimum of 125 seconds.

10.3.2.2. Run the calibration standards as described in Section 10.4. Refer to Appendix 1 for acceptance criteria.

10.3.3. **Determination of Inter-Element Correction Factor (IEC)**

10.3.3.1. Analyze a single element standard using the concentration of the high point in the ICAL.

10.3.3.2. Calculate the IEC factor by dividing the apparent target analyte concentration to the actual concentration of the interference check solution found in the analysis.

10.3.3.3. If the IEC factor is greater than ½ LOQ, update the IEC factor. Otherwise, retain the old IEC.

10.3.3.4. Follow the same procedure for uncommon elements that may contribute spectral interference.

10.3.3.5. Perform IEC determination:

- every six months; OR
- when major instrumentation maintenance is performed (e.g., cleanup/changes of torch, nebulizer, injector, or plasma conditions); OR
- whenever the non-spiked analytes in the ICSA is more than the required level per project specific requirement.

10.3.3.6. When new IEC factor is being used, document the change in the analytical run log.

10.3.3.7. Keep a record of the IEC calculations and IEC Factors for easy reference.

10.3.4. **Establishing Instrument Detection Limit (IDL)**

10.3.4.1. Analyze a minimum of seven consecutive method blanks.

10.3.4.2. Repeat the process within three non-consecutive days.

10.3.4.3. Calculate the standard deviation of each run.

10.3.4.4. The average of the standard deviation of the three runs determines the IDL for each analyte.

10.3.5. **Verifying Linear Dynamic Range (LDR)**

10.3.5.1. 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 ± 10% of the expected value. If non-compliant determine the probable cause. Refer to Section 12 for corrective action.

10.3.5.2. At a minimum perform LDR verification every six months.

10.4. **Analysis**

10.4.1. **Analytical Sequence.**

10.4.1.1. Input the analytical sequence into the instrument using data acquisition software. Refer to Table 8.

10.4.1.2. Set QC limits on QC samples for easy verification while analytical samples are on the run.

10.4.1.3. Using the analytical sequence, arrange the standards and the digestates to be analyzed chronologically using S_0.

10.4.1.4. Transfer about 5 mL of its content into autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence.

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 unspiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.

10.4.1.7. Add 0.025 mL of 1000 mg/L Sc internal standard into each sample.

10.4.1.8. Set the prepared analytical samples into the autosampler and start the analytical run.

10.4.2. **Sample Result Evaluation**

10.4.2.1. Check QC parameters as soon as data is available.

- Check the initial calibration verification (ICV 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. Intensities should be between 50-150%.
- If any of the above checkpoints is non-compliant, perform the specified corrective action in /Appendix 1. If results indicate digestion problem, fill-up an NCR and order re-digestion for the affected sample(s). 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, CCB against Appendix 1.

10.5. **Calculations**

10.5.1. The computer software is designed to calculate the concentrations in the digestates, based on the assumption that the initial calibration is linear through the origin. Thus, for aqueous samples, the computer-produced results represent the concentration of the sample.

10.5.2. For water samples, if the initial sample taken was V_s, different from 100 ml, then calculate the concentration using the following equation:

$$C_s = C_i \left(\frac{V_e}{V_s} \right) DF \qquad \textit{Eq-10.6.2}$$

where:
C_s - concentration in the sample, mg/L
C_i - concentration in the digestate, mg/L
V_s - volume of sample taken, ml
V_e - volume of digestate, ml
DF - dilution factor

10.5.3. For solids, use the following equation to calculate the concentration.

$$C_s = C_i \left(\frac{V_e}{W_s} \right) \left(\frac{100}{100 - \%H_2O} \right) DF \qquad \textit{Eq-10.6.3}$$

where:
C_s – concentration in the sample, mg/Kg
C_i – concentration in the digestate (computer generated), mg/Kg
W_s – sample amount taken, g
V_e – volume of digestate, ml
$\%H_2O$ – percent moisture of the sample
DF – dilution factor

10.5.4. Calculate the percent recovery (%R)

$$\%\ \mathrm{Recovery} = \frac{C_f - C}{C_s} * 100 \qquad \textit{Eq-10.6.4}$$

where:
C_f – concentration found
C – concentration of sample
C_s – concentration of spike

10.5.5. Relative Percent Difference (%RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2} \right)} \times 100 \qquad \textit{Eq-10.6.5}$$

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. **Report Generation**

10.6.1. Print the summary of the analytical run, perform a data transfer into a disk, and convert the instrument electronic output file into an ASCII file format.

10.6.2. Run the ICAPCHK.exe program for calibration check.

10.6.3. Identify samples that need to be re-analyzed, if any, and report all samples that met the analytical requirements.

10.6.4. Generate the report using the following in-house reporting program:

Executable Files	Required Support Files	Output
WDBX[1].exe	Login File (requires network), project.pln, seq_name.sq, seq_name.ckv, seq_name.out	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)
IQCVX.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

10.6.5. Arrange the analysis package in sequence as detailed below. Attach all raw data to every form generated and re-analyses.

- Sample Results
- LCS Summary
- MS Summary
- Duplicate Summary
- CCV Summary
- ICV Summary
- ICAL Summary

10.6.6. Generate the case narrative using CN2.exe.

10.7. **Data Review**

10.7.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- Review the ICAPCHK.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.

[1] X – latest program version

- Verify interference, check results to be within acceptance limits.

10.7.2. Review the case narrative and edit as necessary to reflect essential issues not captured by the case narrative generator program.

10.7.3. If any of the above checkpoints is non-compliant, re-analysis required.

10.8. **Preventive Maintenance**

10.8.1. Refer to Form 6010FM for daily routine maintenance check points.

10.8.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back to control.

10.8.3. 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 injection line. Check autosampler response.	Daily prior to analysis
Verification	Check instrument parameters to ensure normal operating conditions. Change tubings as necessary Check instrument performance (e.g., ICV/ICB)	Daily prior to analysis
Documentation	Record maintenance in instrument service logs	Daily prior to analysis
System Cleaning	Remove covers and clean dust from fans and vent covers	Every 6 months or as necessary
Inspection	Perform general inspection of the complete system	Once a year

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 lots of all reagents.

11.1.2. 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.

11.1.3. All reagents shall be subjected to QC check prior to use. Refer to EMAX-QC01.

11.2. **Sample Analysis QC**

11.2.1. Every analytical run shall be preceded with an initial calibration and initial calibration verification (ICV). Obtain the ICV standard from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis shall be valid unless acceptance criteria are met.

11.2.2. Verify inter-element and background correction factors with ICSA and ICSAB standards after ICB.

11.2.3. 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.

11.2.4. Analyze matrix QC samples, MS/MSD/Dilution Test & Post Digestion spike.

11.2.5. If MS/MSD failed, evaluate Dilution Test for analytes detected ~10x LOQ, evaluate post digestion spike result for ND analytes.

11.2.6. Refer to Appendix 1 for acceptance criteria.

11.3. **Method QC**

11.3.1. Detection Limit, LOD & LOQ must be established before the analytical procedure can be used.

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. Verify LDR 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.

11.3.5. Perform Limit of Detection Verification (LODV) and Limit of Quantitation Verification (LOQV) quarterly.

11.3.6. Perform Inter-Element Corrections (IEC) verifications every six months or whenever there is a significant instrument change.

11.3.7. Perform Instrument Detection Limit (IDL) quarterly.

12.0 CORRECTIVE ACTION

12.1. Quality control procedures and corresponding corrective actions are summarized in Appendix 1.

12.2. 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 or inability to light the plasma is a symptom of a poor drain tube connection
  - Poor precision and carry-over problem 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. Lost 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.

12.3. When ICV is non-compliant consider the following options to correct the problem prior to recalibration:

- Check the gas flow if it is conforming to the instrument parameter setting and correct, if necessary.

- Check the pump tubings and replace, if necessary.
- Check the nebulizer for clogs and clean, if necessary.
- Prepare new standards and re-calibrate.

12.4. When ICB/CCB is non-compliant, consider the following suggestions to correct the problem:

- Replace the instrument blank (S0) with a fresh reagent water and re-analyze the blank.
- If problem persists, check the pump tubings and the nebulizer for clogs and perform maintenance as necessary.

12.5. When CCV is non-compliant, consider the following suggestions to correct the problem:

- Check the connections prior to re-running the ICAL.
- Prepare a new standard and repeat the ICAL.

12.6. When Method Blank (MB) is non-compliant, consider the following suggestions to correct the problem:

- Check if the associated samples detected the same analytes as detected in the MB.
- If analytes detected on the MB is not detected in the associated samples, report the results and flag the analytes detected on the MB as required by the project.
- If similar analytes are detected at concentration levels greater than 10X the detection limits, refer the issue to the PM. If the result is not reportable, check the source of contamination, correct the problem and re-digest all associated samples.
- Prepare and analyze a reagent blank to contain the same acid strength as the digested method blank. If contamination exists, check the reagent to identify where the contamination is coming from. Use new reagents as necessary and re-digest/re-analyze all associated samples.

12.7. 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, Ba, Pb, and Cr, indicating stock standard degradation are as follows:

  Low Silver (Ag) recovery is indicative of chloride contamination causing AgCl precipitation.

  Low Arsenic (As) recovery is indicative of loss during sample preparation as volatile oxides (AsO_3) or precipitation as $AsCl_3$

  Low Barium (Ba) recovery is indicative of SO_4 or CrO_4 contamination. Barium will form precipitates with HF and H_2SO_4.

  High Lead (Pb) recovery is indicative of environmental contamination.

12.8. When MS/MSD is non-compliant verify spike concentrations. If samples are not properly spiked, re-digest and re-analyze. If samples were properly spiked, evaluate post spike and dilution test:

- If parent sample result is “ND”, evaluate post spike.

- If parent sample result is high (i.e., 4x of spike concentration) and post spike failed, evaluate dilution test.
- If results are indicative of matrix interference, discuss in the case narrative.

12.9. When LDR verification is non-compliant consider the following suggestions to correct the problem.

- If all results are bias low or bias high it is indicative of standard preparation error. Prepare a fresh LDR check standard and re-analyze.
- Check the bracketing calibration verifications. If results are indicative of instrument contamination refer to MB refer to ICB/CCB corrective action. If results are indicative of unsuccessful/borderline recovery, refer to ICV/CCV corrective action.
- Check the non-compliant analyte(s) and rule out probable IEC factors. If interfering analyte is a suspect, perform IEC as describe in 10.3.3.

12.10. A Non-Conformance Report (NCR) is required when the following circumstances occur:

12.10.1. Corrective action needs the assistance of the Project Manager, e.g. sample passed the holding time, insufficient amount of sample, etc.

12.10.2. Corrective action prescribed in the IQC Summary does not correct the problem.

12.10.3. Refer to EMAX-QA08 for details of completing an NCR.

12.11. For other problems encountered, inform the supervisor immediately for further instruction.

13.0 POLLUTION PREVENTION

13.1. Unused samples, digestates and instrument wastes are very acidic and are very corrosive. Endorse the waste to the Waste Management Unit for proper treatment or disposal.

14.0 WASTE MANAGEMENT

14.1. Endorse all unused samples, expired analytical standards and other waste generated during the analytical process to WDU shall be disposed 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.

15.1.1.2. **Analytical batch** is compose 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 defined as an instrument response per unit measure. It is an experimental value by measuring the response of an instrument per unit target analyte under the method specific condition. A determinant measured from a standard to obtain the correct value of an instrument output.

15.1.3. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.4. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.5. 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.

15.1.6. Matrix – is a physical state of a sample. Most of environmental samples are classified as water, soil or air.

15.1.7. 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.

15.1.8. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

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. 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.

15.1.11. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

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 QC Procedures**

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Metals Analysis by ICP 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 Evaluating Solid Waste, Physical and Chemical Methods, US EPA SW-846, Method 6010B, Rev.2, Dec. 1996.

16.2. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES

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Table 1: ICP ELEMENTS AND WAVELENGTHS

ELEMENT	SYMBOL	WAVELENGTH
Aluminum	Al	308.215
Antimony	Sb	206.833
Arsenic	As	189.042
Barium	Ba	493.403
Beryllium	Be	313.042
Boron	B	249.773
Cadmium	Cd	226.502
Calcium	Ca	317.933
Chromium	Cr	267.716
Cobalt	Co	228.616
Copper	Cu	324.754
Iron	Fe	271.441
Lead	Pb	220.353
Lithium	Li	670.784
Magnesium	Mg	279.079
Manganese	Mn	257.610
Molybdenum	Mo	202.030
Nickel	Ni	231.604
Potassium	K	766.490
Selenium	Se	196.090
Silver	Ag	328.068
Sodium	Na	589.592
Strontium	Sr	421.552
Thallium	Tl	190.856
Tin	Sn	189.989
Titanium	Ti	334.941
Uranium	U	409.014
Vanadium	V	292.402
Zinc	Zn	206.200

Table 2: CERTIFIED STOCK STANDARD SOLUTIONS

SOURCE	STANDARD	ELEMENTS	CONC. (mg/L)	MATRIX
Accu Standard	EMAX-6010C-1	Be, Cd, Pb, Mn	1000	2-5% HNO3 with trace of HF
Accu Standard	EMAX-6010C-2	Ba, Cu, V	1000	2-5% HNO3 with trace of HF
		Co	200	
Accu Standard	EMAX-6010C-3	B, Mo	1000	2-5% HNO3 with trace of HF
Accu Standard	EMAX-6010C-4	Cr, Ni	1000	2-5% HNO3 with trace of HF
		Ca, K	5000	
Accu Standard	EMAX-6010C-5	Sb, Tl	1000	2-5% HNO3 with trace of HF
Accu Standard	EMAX-6010C-6	Li, Ag, Sr, Sn, Ti	1000	2-5% HNO3 with trace of HF
Accu Standard	EMAX-6010C-6A	As, Se	1000	2-5% HNO3 with trace of HF
Accu Standard	EMAX-6010C-MRL	Al, Mg	200	2-5% HNO3 with trace of HF
		Sb	50	
		As, Cr, Cu, Se, Ag, Tl, V, Ni, Zn, Co, Mo	10	
		Ba, Cd, Mn	5	
		Be	4	
		B, Fe	100	
		Ca, Na	500	
		Pb	3	
		K	1000	
Accu Standard	EMAX-6010C-CCV	Sb, V	150	2-5% HNO3 with trace of HF
		As, Co, Se	100	
		Ba, Cd	200	
		Be, B, Cr, Cu, Mn, Mo, Ni, Tl, Zn, Pb	250	
		Mg, Na, K	6000	
		Li, Ag, Sr, Sn, Ti	50	
CPI	EMAX-6010C-ICV	Sb, V	150	2-5% HNO3 with trace of HF
		As, Co, Se	100	
		Ba, Cd	200	
		Be, B, Cr, Cu, Mn, Mo, Ni, Tl, Zn, Pb	250	
		Mg, Na, K	6000	
		Li, Ag, Sr, Sn, Ti	50	
Accu Standard	EMAX-6010C-ICSA	Al	4000	2-5% HNO3 with trace of HF
		Ca	5000	
		Fe, Mg	2000	
Accu Standard	EMAX-6010C-ICSAB	Sb, As, Cd, Pb, Li, Mo, Ni, Se, Ag, Tl, Sn, Ti, Zn	100	2-5% HNO3 with trace of HF
		Ba, Be, B, Cr, Co, Cu, Mn, Sr, V	50	
		Na, K	7500	

Table 3: INITIAL CALIBRATION STANDARDS PREPARATION

Standard Name	Vendor	Standard Source	Analytes	Conc. (mg/L)	Aliquot (mL)	Final Conc. (mg/L)
S0	Reagent Water	So		0	1000	0
S20	Accu Standard	EMAX-6010C-1	Be, Cd, Pb, Mn	1000	1	1
S21	Accu Standard	EMAX-6010C-1	Be, Cd, Pb, Mn	1000	10	10
S22	Accu Standard	EMAX-6010C-2	Ba, Cu, V	1000	1	1
			Co	200		0.2
	Accu Standard	Single Analyte	Fe	10000	1	10
S23	Accu Standard	EMAX-6010C-2	Ba, Cu, V	1000	10	10
			Co	200		2
	Accu Standard	Single Standard	Fe	10000	40	400
S24	Accu Standard	EMAX-6010C-3	B, Mo	1000	1	1
	Accu Standard	Single Standard	Na	10000	5	50
S25	Accu Standard	EMAX-6010C-3	B, Mo	1000	10	10
	Accu Standard	Single Standard	Na	10000	30	300
S26	Accu Standard	EMAX-6010C-4	Cr, Ni	1000	1	1
			Ca, K	5000		50
	Accu Standard	Single Standard	Al	10000	1	10
S27	Accu Standard	EMAX-6010C-4	Cr, Ni	1000	10	10
			Ca, K	5000		500
	Accu Standard	Single Standard	Al	10000	40	400
S28	Accu Standard	EMAX-6010C-5	Sb, Tl	1000	1	1
	Accu Standard	Single Standard	Mg	10000	5	50
S29	Accu Standard	EMAX-6010C-5	Sb, Tl	1000	10	10
	Accu Standard	Single Standard	Mg	10000	20	200
S30	Accu Standard	EMAX-6010C-6	Li, Ag, Sr, Sn, Ti	100	2	0.2
	Accu Standard	EMAX-6010C-6A	As, Se	100	5	0.5
	Accu Standard	Single Standard	Zn	1000	1	1
S31	Accu Standard	EMAX-6010C-6	Li, Ag, Sr, Sn, Ti	100	10	1
	Accu Standard	EMAX-6010C-6A	As, Se	100	20	2
	Accu Standard	Single Standard	Zn	1000	10	10

Table 3A: INITIAL CALIBRATION STANDARDS CONCENTRATION

Element	Concentration (mg/L)		
	S0	ICAL Pt. 1	ICAP Pt. 2
Al	0	10	400
Sb	0	1	10
As	0	0.5	2
Ba	0	1	10
Be	0	1	10
B	0	1	10
Cd	0	1	10
Ca	0	50	500
Cr	0	1	10
Co	0	0.2	2
Cu	0	1	10
Fe	0	10	400
Pb	0	1	10
Li	0	0.2	1
Mg	0	50	200
Mn	0	1	10
Mo	0	1	10
Ni	0	1	10
K	0	50	500
Se	0	0.5	2
Ag	0	0.2	1
Na	0	50	300
Sr	0	0.2	1
Tl	0	1	10
Sn	0	0.2	1
Ti	0	0.2	1
V	0	1	10
Zn	0	1	10

Table 4: INITIAL CALIBRATION VERIFICATION STANDARD PREPARATION

Standard Name	Vendor	Standard Source	Analytes	Conc. (mg/L)	Aliquot (mL)	Final Conc. (mg/L)
ICV	CPI	EMAX-6010C-ICV	Sb, V, Be	150	1	1.5
			As, Co, Se	100		1
			Ba, Cd	200		2
			B, Cr, Cu, Mn, Mo, Ni, Tl, Zn, Pb	250		2.5
			Mg, Na, K	6000		60
			Li, Ag, Sr, Sn, Ti	50		0.5
	CPI	Single Analyte	Ca	10000	0.75	75
	CPI	Single Analyte	Al	10000	1	100
	CPI	Single Analyte	Fe	10000	0.25	25

Table 5. CONTINUING CALIBRATION VERIFICATION STANDARD PREPARATION

Standard Name	Vendor	Standard Source	Analytes	Conc. (mg/L)	Aliquot (mL)	Final Conc. (mg/L)
CCV	Accu Standard	EMAX-6010C-CCV	Sb, V, Be	150	1	1.5
			As, Co, Se	100		1
			Ba, Cd	200		2
			B, Cr, Cu, Mn, Ni, Tl, Zn, Pb	250		2.5
			Mg, Na, K	6000		60
			Li, Ag, Sr, Sn, Ti	50		0.5
	Accu Standard	Single Analyte	Ca	10000	0.75	75
	Accu Standard	Single Analyte	Al	10000	1	100
	Accu Standard	Single Analyte	Fe	10000	0.25	25

Table 6: ICSA AND ICSAB STANDARDS PREPARATION

Standard Name	Vendor	Standard Source	Analytes	Conc. (mg/L)	Aliquot (mL)	Final Conc. (mg/L)
ICSA	Accu Standard	EMAX-6010C-ICSA	Al	4000	100	400
			Ca	5000		500
			Fe, Mg	2000		200
ICSAB	Accu Standard	EMAX-6010C-ICSAB	Sb, As, Cd, Pb, Li, Mo, Ni, Se, Ag, Tl, Sn, Ti, Zn	100	10	1
			Ba, Be, B, Cr, Co, Cu, Mn, Sr, V	50		0.5
			Na, K	7500		75
	Accu Standard	EMAX-6010C-ICSA	Al	4000	100	400
			Ca	5000		500
			Fe, Mg	2000		200

Table 7: ICP DETECTION LIMITS

ELEMENT	WATER (mg/L)			SOIL (mg/Kg)		
	DL	LOD	LOQ	DL	LOD	LOQ
Aluminum	0.0112	0.03	0.2	1.6114	5	20
Antimony	0.0032	0.03	0.1	0.2764	3	10
Arsenic	0.0030	0.005	0.01	0.1180	0.4	1
Barium	0.0013	0.002	0.01	0.0981	0.2	1
Beryllium	0.00017	0.0005	0.01	0.0339	0.1	1
Boron	0.0031	0.01	0.1	0.2854	1	10
Cadmium	0.0003	0.002	0.01	0.0264	0.2	1
Calcium	0.0766	0.1	1	9.9366	10	100
Chromium	0.00077	0.003	0.01	0.0361	0.3	1
Cobalt	0.00058	0.002	0.01	0.04	0.2	1
Copper	0.00075	0.003	0.01	0.044	0.3	1
Iron	0.011	0.04	0.2	0.714	3	20
Lead	0.0017	0.003	0.01	0.09	0.3	1
Lithium	0.0062	0.05	1	0.119	0.5	2
Magnesium	0.0268	0.1	1	5.4602	10	100
Manganese	0.0005	0.003	0.01	0.043	0.3	1
Molybdenum	0.0005	0.003	0.01	0.05	0.3	5
Nickel	0.0011	0.003	0.01	0.049	0.3	1
Potassium	0.0712	0.1	1	2.8020	10	100
Selenium	0.0018	0.005	0.01	0.27	0.5	1
Silver	0.0004	0.003	0.01	0.0309	0.3	1
Sodium	0.036	0.1	1	2.904	10	100
Strontium	0.0008	0.001	0.01	0.050	0.1	1
Thallium	0.0010	0.005	0.01	0.051	0.5	1
Tin	0.0009	0.005	1	1.1926	10	25
Titanium	0.0009	0.005	1	0.13	0.5	10
Vanadium	0.0005	0.002	0.01	0.0430	0.15	1
Zinc	0.0072	0.01	0.020	0.61	1	2

Table 8: ICP ANALYTICAL SEQUENCE FOR 6010 METHOD

RUN ID LABEL	SAMPLE DESCRIPTION	SOLUTION ID LABEL
S0	Calibration Standard 1 (blank)	S0
S20-S31	ICAL - STDS	S20-S31
ICV	Initial Calibration Verification	ICV
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
IPMSSSXB	Preparation Blank	
IPMSSSXL/C	Lab Control Sample	
Sample 1	Sample 1	
Sample 1M	Sample 1 MS Duplicate	
Sample 1S	Sample 1 Matrix Spike	
Sample 1J	Sample 1 Serial Dilution(5 x dilution sample 1)	
Sample 1A	Sample 1 Post Digestion spike	
Sample 2 to 4	Sample 2 to Sample 5	
CCV2	Continuing Calibration Verification #2	CCV
CCB2	Continuing Calibration Blank #2	S0
Sample 5 to 14	Maximum of 10 Samples	
CCV3	Continuing Calibration Verification #3	CCV
CCB3	Continuing Calibration Blank #3	S0
Sample 15 to 20	Sample 15 to 20 or a maximum of 10 samples (sample 15 to 24)	
CCV4	Continuing Calibration Verification #4	CCV
CCB4	Continuing Calibration Blank #4	S0

Figure 1: **TYPICAL SAMPLE REPORT**

METHOD 3010A/6010B
METALS BY TRACE ICP

Client :	XYZ, INC.	Date Collected:	06/30/10 08:25
Project :	CLEAN PROJECT	Date Received:	06/30/10
SDG NO. :	10F352	Date Extracted:	07/01/10 10:10
Sample ID:	1D-T-1EF-30JUN10	Date Analyzed:	07/02/10 18:36
Lab Samp ID:	F352-01	Dilution Factor:	1
Lab File ID:	ID8G004031	Matrix :	WATER
Ext Btch ID:	IPG001W	% Moisture :	NA
Calib. Ref.:	ID8G004025	Instrument ID :	EMAXTID8

PARAMETERS	RESULTS (mg/L)	LOQ (mg/L)	LOD (mg/L)
Aluminum	0.155J	0.200	0.0600
Antimony	ND	0.100	0.0100
Arsenic	1.21	0.0100	0.00500
Barium	0.143	0.0100	0.00200
Beryllium	ND	0.0100	0.00100
Cadmium	ND	0.0100	0.00100
Calcium	23.3	1.00	0.100
Chromium	ND	0.0100	0.00250
Cobalt	ND	0.0100	0.00250
Copper	ND	0.0100	0.00200
Iron	2.18	0.200	0.0400
Lead	0.00393J	0.0100	0.00300
Magnesium	16.6	1.00	0.100
Manganese	4.29	0.0100	0.00300
Nickel	ND	0.0100	0.00250
Potassium	1.47	1.00	0.100
Selenium	ND	0.0100	0.00500
Silver	ND	0.0100	0.00300
Sodium	86.1	1.00	0.100
Thallium	0.00765J	0.0100	0.00500
Vanadium	ND	0.0100	0.00500
Zinc	ND	0.0100	0.00500

Figure 2: **TYPICAL LCS/LCD SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN PROJECT
SDG NO.: 10F352
METHOD: METHOD 3010A/6010B

MATRIX:	WATER			% MOISTURE:	NA
DILTN FACTR:	1	1	1		
SAMPLE ID:	MBLK1W				
CONTROL NO.:	IPG001WB	IPG001WL	IPG001WC		
LAB FILE ID:	ID8G004027	ID8G004028	ID8G004029		
DATIME EXTRCTD:	07/01/1010:10	07/01/1010:10	07/01/1010:10	DATE COLLECTED:	NA
DATIME ANALYZD:	07/02/1018:17	07/02/1018:22	07/02/1018:27	DATE RECEIVED:	07/01/10
PREP. BATCH:	IPG001W	IPG001W	IPG001W		
CALIB. REF:	ID8G004025	ID8G004025	ID8G004025		

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 %
Aluminum	ND	5	4.93	98	5	4.9	98	0	80-120	20
Antimony	ND	2.5	2.73	109	2.5	2.72	109	0	80-120	20
Arsenic	ND	.5	.533	107	.5	.529	106	1	80-120	20
Barium	ND	.5	.472	94	.5	.471	94	0	80-120	20
Beryllium	ND	.5	.506	101	.5	.504	101	0	80-120	20
Cadmium	ND	.5	.516	103	.5	.512	102	1	80-120	20
Calcium	ND	50	51.2	102	50	50.9	102	0	80-120	20
Chromium	ND	.5	.478	96	.5	.477	95	0	80-120	20
Cobalt	ND	.5	.512	102	.5	.511	102	0	80-120	20
Copper	ND	.5	.497	99	.5	.492	98	1	80-120	20
Iron	ND	5	5.03	101	5	4.96	99	1	80-120	20
Lead	ND	.5	.495	99	.5	.492	98	1	80-120	20
Magnesium	ND	50	51.7	103	50	51.8	104	0	80-120	20
Manganese	ND	.5	.488	98	.5	.487	97	0	80-120	20
Nickel	ND	.5	.503	101	.5	.499	100	1	80-120	20
Potassium	ND	50	50.5	101	50	50.4	101	0	80-120	20
Selenium	ND	.5	.535	107	.5	.533	107	0	80-120	20
Silver	ND	.5	.511	102	.5	.511	102	0	80-120	20
Sodium	ND	50	50.3	101	50	50.4	101	0	80-120	20
Thallium	ND	.5	.478	96	.5	.473	95	1	80-120	20
Vanadium	ND	.5	.509	102	.5	.509	102	0	80-120	20
Zinc	ND	.5	.533	107	.5	.528	106	1	80-120	20

Figure 3: **TYPICAL MATRIX SPIKE SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN PROJECT
SDG NO.: 10F352
METHOD: METHOD 3010A/6010B

MATRIX:	WATER			% MOISTURE:	NA
DILTN FACTR:	1	1	1		
SAMPLE ID:	1D-T-1EF-30JUN10				
CONTROL NO.:	F352-01	F352-01M	F352-01S		
LAB FILE ID:	ID8G004031	ID8G004033	ID8G004034		
DATIME EXTRCTD:	07/01/1010:10	07/01/1010:10	07/01/1010:10	DATE COLLECTED:	06/30/10 08:25
DATIME ANALYZD:	07/02/1018:36	07/02/1018:46	07/02/1018:51	DATE RECEIVED:	06/30/10
PREP. BATCH:	IPG001W	IPG001W	IPG001W		
CALIB. REF:	ID8G004025	ID8G004025	ID8G004025		

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 %
Aluminum	.155J	5	5.11	99	5	5.06	98	1	75-125	20
Antimony	ND	2.5	2.89	116	2.5	2.9	116	0	75-125	20
Arsenic	1.21	.5	1.75	109	.5	1.76	110	0	75-125	20
Barium	.143	.5	.621	96	.5	.618	95	0	75-125	20
Beryllium	ND	.5	.518	104	.5	.516	103	0	75-125	20
Cadmium	ND	.5	.531	106	.5	.529	106	0	75-125	20
Calcium	23.3	50	76.5	106	50	76	105	1	75-125	20
Chromium	ND	.5	.485	97	.5	.483	97	0	75-125	20
Cobalt	ND	.5	.529	106	.5	.528	106	0	75-125	20
Copper	ND	.5	.506	101	.5	.5	100	1	75-125	20
Iron	2.18	5	7.2	100	5	7.13	99	1	75-125	20
Lead	.00393J	.5	.507	101	.5	.504	100	1	75-125	20
Magnesium	16.6	50	70.7	108	50	70.8	108	0	75-125	20
Manganese	4.29	.5	4.66	72*	.5	4.64	69*	0	75-125	20
Nickel	ND	.5	.516	103	.5	.514	103	0	75-125	20
Potassium	1.47	50	55	107	50	55.1	107	0	75-125	20
Selenium	ND	.5	.539	108	.5	.541	108	0	75-125	20
Silver	ND	.5	.524	105	.5	.521	104	1	75-125	20
Sodium	86.1	50	136	100	50	135	98	1	75-125	20
Thallium	.00765J	.5	.483	95	.5	.477	94	1	75-125	20
Vanadium	ND	.5	.521	104	.5	.52	104	0	75-125	20
Zinc	ND	.5	.548	110	.5	.545	109	1	75-125	20

Figure 4: **TYPICAL SERIAL DILUTION TEST REPORT**

EMAX QUALITY CONTROL DATA
SERIAL DILUTION ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN PROJECT
BATCH NO.: 10F352
METHOD: METHOD 3010A/6010B

MATRIX:	WATER		% MOISTURE:	NA
DILUTION FACTOR:	1	5		
SAMPLE ID:	1D-T-1EF-30JUN10	1D-T-1EF-30JUN10DL		
EMAX SAMP ID:	F352-01	F352-01J		
LAB FILE ID:	ID8G004031	ID8G004032		
DATE EXTRACTED:	07/01/1010:10	07/01/1010:10	DATE COLLECTED:	06/30/10 08:25
DATE ANALYZED:	07/02/1018:36	07/02/1018:41	DATE RECEIVED:	06/30/10
PREP. BATCH:	IPG001W	IPG001W		
CALIB. REF:	ID8G004025	ID8G004025		

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	SERIAL DIL RSLT (mg/L)	DIF RSLT %	QC LIMIT (%)
Aluminum	0.155J	ND	NA	10
Antimony	ND	ND	0	10
Arsenic	1.21	1.17	3	10
Barium	0.143	0.144	0	10
Beryllium	ND	ND	0	10
Cadmium	ND	ND	0	10
Calcium	23.3	23.6	1	10
Chromium	ND	ND	0	10
Cobalt	ND	ND	0	10
Copper	ND	ND	0	10
Iron	2.18	2.24	3	10
Lead	0.00393J	ND	NA	10
Magnesium	16.6	16.8	1	10
Manganese	4.29	4.37	2	10
Nickel	ND	ND	0	10
Potassium	1.47	1.46J	NA	10
Selenium	ND	ND	0	10
Silver	ND	ND	0	10
Sodium	86.1	85.0	1	10
Thallium	0.00765J	ND	NA	10
Vanadium	ND	ND	0	10
Zinc	ND	ND	0	10

Figure 5: **TYPICAL ANALYTICAL SPIKE REPORT**

EMAX QUALITY CONTROL DATA
ANALYTICAL SPIKE ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN PROJECT
SDG NO.: 10F352
METHOD: METHOD 3010A/6010B

MATRIX:	WATER		% MOISTURE:	NA
DILTN FACTR:	1	1		
SAMPLE ID:	1D-T-1EF-30JUN10			
CONTROL NO.:	F352-01	F352-01A		
LAB FILE ID:	ID8G004031	ID8G004030		
DATIME EXTRCTD:	07/01/1010:10	07/01/1010:10	DATE COLLECTED:	06/30/10 08:25
DATIME ANALYZD:	07/02/1018:36	07/02/1018:31	DATE RECEIVED:	06/30/10
PREP. BATCH:	IPG001W	IPG001W		
CALIB. REF:	ID8G004025	ID8G004025		

ACCESSION:

PARAMETER	SMPL RSLT (mg/L)	SPIKE AMT (mg/L)	AS RSLT (mg/L)	AS % REC	QC LIMIT (%)
Aluminum	.155J	5	4.78	93	75-125
Antimony	ND	2.5	2.68	107	75-125
Arsenic	1.21	.5	1.69	96	75-125
Barium	.143	.5	.603	92	75-125
Beryllium	ND	.5	.498	100	75-125
Cadmium	ND	.5	.515	103	75-125
Calcium	23.3	50	72.8	99	75-125
Chromium	ND	.5	.468	94	75-125
Cobalt	ND	.5	.516	103	75-125
Copper	ND	.5	.479	96	75-125
Iron	2.18	5	6.86	94	75-125
Lead	.00393J	.5	.485	96	75-125
Magnesium	16.6	50	67.3	101	75-125
Manganese	4.29	.5	4.56	52*	75-125
Nickel	ND	.5	.5	100	75-125
Potassium	1.47	50	52.4	102	75-125
Selenium	ND	.5	.501	100	75-125
Silver	ND	.5	.473	95	75-125
Sodium	86.1	50	133	93	75-125
Thallium	.00765J	.5	.462	91	75-125
Vanadium	ND	.5	.502	100	75-125
Zinc	ND	.5	.534	107	75-125

Figure 6: **TYPICAL CASE NARRATIVE**

CASE NARRATIVE

Client : XYZ, INC

Project : CLEAN PROJECT

SDG : 10F352

METHOD 3010A/6010B
METALS BY TRACE ICP

A total of four (4) water samples were received on 06/30/10 for Metals TAL by ICP analysis, Method 3010A/6010B 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. MRL was analyzed as required by the project.

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 IPG001WL/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 one result qualified with [*] in F352-01 M/S summary form, most likely due to matrix interference.
Check QC summary 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	1st Rvw	2nd Rvw
Initial Calibration (min. 1 pt std and a blank)	Daily initial calibration prior to sample analysis	Acceptable ICV	Correct the problem and repeat initial calibration.		
Initial Calibration Verifications (ICV) Second Source	Daily after initial calibration.	All analytes within ± 10% of expected value RSD of Replicate integrations: < 5%	Correct the problem and repeat initial calibration.		
Calibration Verifications (CCV)	Daily before sample analysis, 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	No analytes detected ≥ 3X IDL	Correct problem then reanalyze calibration blank and previous samples.		
Interference Check Sample (ICSA/ICSAB)	Analyzed at the beginning of each analytical run	Within ± 20% of expected value	Terminate analysis, correct the problem, reanalyze ICS, and reanalyze all affected samples		
Method Blank	One per preparation batch	No analytes detected > ½ LOQ	Re-prep and reanalyze method blank and all samples processed with the contaminated blank. Otherwise, apply B to specific analyte(s) on all associated samples		
Laboratory Control Sample (LCS)	One per preparation batch	% Recovery : 80 - 120%	Re-prep and reanalyze LCS and all associated samples		
Sample Duplicate	Per project requirement	RPD ≤ 20%	• If indicative of matrix interference in the absence of PSR, discuss in case narrative		
Matrix Spikes (MS/MSD)	One MS/MSD per preparation batch or as required by the project	% Recovery: 75 – 125% RPD ≤ 20%	Evaluate post spike and dilution test: • If parent sample result is "ND", evaluate post spike. • If parent sample result is high (i.e., 4x of spike concentration) and post spike failed, evaluate dilution test.		
Dilution Test (5X)	One for every MS prepared	1:5 dilution must agree within ± 10% of the original determination	If indicative of matrix interference in the absence of PSR, discuss in case narrative		
Post Digestion Spike Addition	One for every MS prepared	Recovery within 75-125% of expected value	If indicative of matrix interference in the absence of PSR, discuss in case narrative		
Comments: For reporting results between LOD & LOQ, refer to PSR.			Reviewed By		
			Date:		

Appendix 2: **DEMONSTRATION OF CAPABILITY**

PT # **WP0110 Water Pollution Proficiency Testing** **PT Study Closing Date: Feb. 18, 2010**

TRACE METALS

Method: SW 3010 A / SW 6010 B Analyst: Tina Hoang

#	Analyte	Result		Assigned Value	Acceptance Limits			Comments
1	Aluminum	892	µg/L	915	734	-	1090	Acceptable
2	Antimony	316	µg/L	283	192	-	343	Acceptable
3	Arsenic	463	µg/L	445	372	-	522	Acceptable
4	Barium	1650	µg/L	1650	1430	-	1860	Acceptable
5	Beryllium	683	µg/L	662	563	-	747	Acceptable
6	Boron	1170	µg/L	1130	927	-	1310	Acceptable
7	Cadmium	269	µg/L	258	220	-	294	Acceptable
8	Chromium	723	µg/L	718	626	-	811	Acceptable
9	Cobalt	274	µg/L	268	235	-	301	Acceptable
10	Copper	356	µg/L	348	314	-	384	Acceptable
11	Iron	2080	µg/L	2050	1820	-	2310	Acceptable
12	Lead	1730	µg/L	1680	1480	-	1880	Acceptable
13	Manganese	337	µg/L	344	308	-	383	Acceptable
14	Molybdenum	430	µg/L	436	369	-	499	Acceptable
15	Nickel	861	µg/L	844	759	-	943	Acceptable
16	Selenium	336	µg/L	323	254	-	375	Acceptable
17	Silver	238	µg/L	228	196	-	262	Acceptable
18	Strontium	105	µg/L	106	91.5	-	122	Acceptable
19	Thallium	399	µg/L	385	302	-	468	Acceptable
20	Vanadium	510	µg/L	506	443	-	566	Acceptable
21	Zinc	1460	µg/L	1430	1230	-	1640	Acceptable

TIN AND TITANIUM

Method: SW 3010 A / SW 6010 B Analyst: Tina Hoang

#	Analyte	Result		Assigned Value	Acceptance Limits			Comments
1	Tin	3520	µg/L	3650	2870	-	4440	Acceptable
2	Titanium	115	µg/L	122	104	-	138	Acceptable

MINERALS

Method: SW 3010 A / SW 6010 B Analyst: Tina Hoang

#	Analyte	Result		Assigned Value	Acceptance Limits			Comments
1	Calcium	38.7	mg/L	37.7	33.6	-	42.9	Acceptable
2	Magnesium	20.2	mg/L	20.4	17.5	-	23.4	Acceptable
3	Potassium	31.2	mg/L	30.1	24.9	-	35.9	Acceptable
4	Sodium	80.9	mg/L	78.7	66.8	-	90.2	Acceptable

Appendix 2 (cont.): **DEMONSTRATION OF CAPABILITY**

PT # HW0110 Soil/Hazardous Waste Proficiency Testing PT Study Closing Date: Mar. 4, 2010

METALS

Method: SW 3050 B / SW 6010 B Analyst: Tina Hoang

#	Analyte	Result		Assigned Value	Acceptance Limits			Comments
1	Aluminum	9860	mg/Kg	13200	3320	-	14500	Acceptable
2	Antimony	38	mg/Kg	158	15.8	-	174	Acceptable
3	Arsenic	224	mg/Kg	238	155	-	281	Acceptable
4	Barium	226	mg/Kg	243	166	-	286	Acceptable
5	Beryllium	175	mg/Kg	195	132	-	218	Acceptable
6	Boron	98	mg/Kg	128	45.4	-	141	Acceptable
7	Cadmium	169	mg/Kg	185	120	-	208	Acceptable
8	Calcium	10000	mg/Kg	12100	6910	-	13300	Acceptable
9	Chromium	102	mg/Kg	104	68.4	-	129	Acceptable
10	Cobalt	142	mg/Kg	163	108	-	182	Acceptable
11	Copper	136	mg/Kg	151	100	-	167	Acceptable
12	Iron	10200	mg/Kg	11800	2500	-	18400	Acceptable
13	Lead	124	mg/Kg	154	96.9	-	170	Acceptable
14	Magnesium	2810	mg/Kg	3580	1880	-	3940	Acceptable
15	Manganese	316	mg/Kg	383	227	-	421	Acceptable
16	Molybdenum	82	mg/Kg	90.9	53	-	101	Acceptable
17	Nickel	181	mg/Kg	192	120	-	211	Acceptable
18	Potassium	3350	mg/Kg	3130	1820	-	4020	Acceptable
19	Selenium	137	mg/Kg	156	85.7	-	172	Acceptable
20	Silver	66	mg/Kg	73.2	41.6	-	82.8	Acceptable
21	Sodium	1980	mg/Kg	2400	1140	-	2640	Acceptable
22	Strontium	285	mg/Kg	315	188	-	347	Acceptable
23	Thallium	106	mg/Kg	126	72.5	-	143	Acceptable
24	Tin	133	mg/Kg	181	78.6	-	199	Acceptable
25	Titanium	141	mg/Kg	97	7.63	-	224	Acceptable
26	Vanadium	134	mg/Kg	142	84.8	-	162	Acceptable
27	Zinc	391	mg/Kg	421	258	-	463	Acceptable

6010FA. **ANALYSIS RUN LOG**

EMAX LABORATORIES, INC

ANALYSIS RUN LOG
for
ICP

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Start Date:

End Date:

SOP #	Rev. #
☐ EMAX-6010	
☐ EMAX-6010C	
☐ EMAX-200.7	
☐ EMAX-	

Comments:

Book #: AD8-016

Instrument No.: D8

Analytical Batch:

Analytical Sequence:

Method File:

Database:

	STANDARDS ID		STANDARDS ID
S0		S30	
S1		S31	
S2		ICV	
S3		CCV	
S4		ICSA	
S5		ICSAB	
S6		CRVMRL	
S7		LLCV	
S8		MLCV	
S9			
S10			
S11			
S12			
S16			
S17			
S18			
S20			
S21			
S22			
S23			
S24			
S25			
S26			
S27			
S28			
S29			

Analyzed By:

Date:

6010FS: **SAMPLE PREPARATION LOG**

EMAX LABORATORIES, INC.

Page 1

DIGESTION LOG FOR ICP

Note: For samples, relevant QCs/Standards digested, refer to attached digestion sequence.

Comments:

Book #: EIP-095

Batch:

Matrix:

SOP #	Rev. #
☐ EMAX-3005	
☐ EMAX-3010	
☐ EMAX-3050	
☐ EMAX-200.7	
☐ EMAX-	

Start	Temp	End	Temp
	°C		°C

Standards	ID	Amount Added (mL)
LCS-1		
LCS-2		
LCS-3		
MS		
Reagent	**Lot# / ID**	**Amount Added (mL)**
HNO_3		
HCl		
H_2O_2		
HNO3(1:1)		
Digestate Location		
Extract Location		

☐ Reagent Water ID:

☐ HNO_3 dispenser checked @ 1.0 ml with Class A volumetric flask

☐ HNO_3 dispenser checked @ 5.0 ml with Class A volumetric flask

☐ HCl dispenser checked @ 2.0 ml with Class A volumetric flask

☐ HCl dispenser checked @ 10.0 ml with Class A volumetric flask

☐ Thermometer ID:

Prepared By:

Standard Added By: Witnessed By:

Extract Rcvd By: Checked By:

Date endorsed to waste Disposal:

Accepted By:

6010FM: **INSTRUMENT MAINTENANCE LOG**

MAINTENANCE LOG
for
ICAP-6500

Page 1

Instrument No: D8 Month: Year: 201_ Book # MD8-004

Date							
Argon/Nitrogen							
Instrument Discharge (below setting marks)							
Pump Tubings							
Rinse Bottle							
Printer Paper							
Cooling Water °F							
RF Power: 750-1350W							
Pump Rate: 0-125 rpm							
Aux. Gas Flow: 0.0 - 2.0 L/min							
Neb. Gas Flow: 0.0 - 1.50 L/min							
Coolant Gas Flow: 10 - 20 L/min							
Neb Pressure kPa							
ICP Profile							
Room Temperature °F							
Gen Temp when plasma is lit °C							
Optics Temp °C							
Camera Temp °C							
Analyst Initials							

Trouble Shooting							
Problem(s) Encountered							
Corrective Action							
Return To Specs (Analyst Initials)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURES

MERCURY IN LIQUID WASTE

SOP No.:	EMAX-7470	Revision No.	8	Effective Date:	13-Jul-16
Prepared By:	Mary Jane Mendoza			Date:	07.12.16
Approved By:	Kenette Pimentel QA Manager			Date:	07.12.16
Approved By:	Caspar Pang Laboratory Director			Date:	07-12-16

Control Number: 7470-08-

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

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 Preservation**

5.1.1. Samples are expected to be contained in HDPE pre-cleaned containers and preserved with HNO3 to pH < 2.

5.2. **Holding Time**

5.2.1. Samples must be analyzed within 28 days from date of collection.

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	Volumetric Labware and Micropipette Verification
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 SDS of 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, 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, Win Hg Runner 1.3

8.1.1.2. Leeman Hydra AA Automated Analyzer with Autosampler, Computer, Printer and PS200 Software, Win Hg Runner 1.3

8.1.2. 100 ml Digestion vessel

8.1.3. Digestion block or equivalent

8.1.4. Micropipettes and tips

8.1.5. 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. Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride in reagent water and dilute to 1 L. *(Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.)*

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 $SnCl_2$ in reagent water, add 200 ml HCl and volume to 2 L.

8.2.8. Reagent water – Mercury-free water

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.

Stock Std	Name	Source	Conc.	Used for
Primary	Mercury	ERA or equivalent	1000 mg/L	ICAL, CCV
Secondary	Mercury	AccuStandard or equivalent	1000 mg/L	ICV/LCS/MS

9.3.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10°C to -20°C.

9.4. **Intermediate Standard Solution**

9.4.1. Primary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg primary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.2. Secondary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg secondary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.3. Post Spike Standard, 100 µg/L: Dilute 1.0 mL of 10 mg/L Hg primary intermediate standard to 100 mL using reagent water. Digest prior to use.

9.5. **Working Standard**

9.5.1. Primary Working Standard, 50 µg/L: Dilute 0.50 mL of 10 mg/L primary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.5.2. Secondary Working Standard, 50 µg/L: Dilute 0.50 mL of 10 mg/L secondary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.6. **Initial Calibration Standards**

9.6.1. Initial calibration consists of five standards and a reagent water blank. Using the primary working standard, prepare the ICAL points in reagent water, as follows:.

Level	Primary Working Standard, 100 µg/L Aliquot (ml)	Final Volume (mL	Concentration (µg/L)
S0	0	50	0
S1	0.2	50	0.2
S2	0.5	50	0.5
S3	1.0	50	1.0
S4	2.0	50	2.0
S5	5.0	50	5.0

Note: Other concentration levels may be prepared as appropriate to meet project quality objectives.

9.7. **Initial Calibration Verification Standard (ICV)**

9.7.1. Prepare ICV at 2 µg/L by diluting 2mL of secondary working standard (50 µg/L) to final volume of 50 mL using reagent water

9.8. **Continuing Calibration Verification Standard (CCV)**

9.8.1. Prepare CCV at 2 µg/L by diluting 2 mL of primary working standard (50 µg/L) to final volume of 50 mL using reagent water.

9.9. **Laboratory Control Standard (LCS) / Matrix Spike (MS) Standard**

9.9.1. Spike 2.5mL of secondary working standard (50 µg/L) to 50 mL reagent water (for LCS) or designated sample (for MS).

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. Prepare LCS/MS (Sec. 9.9.1). Spike appropriate standards for ICAL standards (Sec. 9.6.1), ICV (Sec. 9.7.1) and CCV (Sec. 9.8.1).

10.1.2.1. **Spike Addition**. Call a witness. Have the witness verify the setting of the micropipette and the expiration date of the spike standard.

10.1.3. Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL concentrated HNO_3 with mixing after each addition.

10.1.4. Add 7.5 mL of 5% $KMnO_4$ 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 (maximum total amount of 12.5 mL) swirling the Digestion vessel at every addition, until purple color persists.

Add the same 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 ± 3°C.

10.1.7. Allow the samples to cool.

10.1.8. Add 3 mL NaCl-hydroxylamine hydrochloride solution and wait for the sample to decolorize. Additional NaCl-hydroxylamine hydrochloride solution may be added to reduce excess permanganate in 1 mL increments up to a maximum total amount of 5 mL.

Add the same amount of NaCl-hydroxylamine hypochloride solution added to the method blank, LCS, calibration standards and calibration verification standards.

10.1.9. Upon decolorization, dilute to 80 mL final volume 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

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
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	50 sec.	60 sec.

CALIBRATION S0, S1, S2, S3, S4, S5	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0

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 Sec. 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% $SnCl_2$, 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 Sec. 9.6.1.

10.3.2.2. Digest the ICAL standards as described in Sec. 10.1.

10.3.2.3. Analyze them as described in Sec. 10.4.

10.3.2.4. Refer to Sec. 10.5 for calculation.

10.3.2.5. Initiate initial calibration as described in the instrument operations manual. After calibration is completed, acquire the calibration data and review.

10.3.2.6. Refer to Appendix 1 for acceptance criteria.

10.3.2.7. Verify the initial calibration by a secondary source standard (ICV).

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Prepare ICV as described in Sec. 9.7.1. Prepare ICB using reagent water.

10.3.3.2. Digest ICV and ICB as described in Sec. 10.1.

10.3.3.3. Analyze the ICV sample to verify the concentration of the ICAL. Analyze the ICB after the ICV.

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 Sec. 9.8.1. Prepare CCB using reagent water.

10.3.4.2. Digest the CCV and CCB as described in Sec. 10.1.

10.3.4.3. Analyze CCV sample to verify the validity of ICAL. Analyze the CCB after the CCV.

10.3.4.4. Refer to Appendix 1 for 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**

S0	0.00000
S1	0.20000
S2	0.50000
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 Digestion Spike (Sec. 10.4.4)

10.4.2.9. Parent Sample

10.4.2.10. Serial Dilution (Sec. 10.4.5)

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. Using the analytical sequence, arrange the digested standards and samples to be analyzed chronologically.

10.4.4. 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.

10.4.5. Prepare a Dilution Test sample at 5x dilution. Pipette 2 mL of sample, add 8 mL of S0 into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.

10.4.6. Prepare a Post Digestion Spike test sample. Using 10 mL unspiked sample digestate (preferably the QC sample), add 0.3 mL Post Spike standard (Sec. 9.4.3).

10.4.7. Set the prepared samples into the autosampler and start the analytical run.

10.4.8. Sample Result Evaluation

10.4.8.1. Check the QC data as soon as available.

- ✓ Check the ICAL and ICV prior to sample analysis.
- ✓ Check the method blank and LCS against project specific requirements.
- ✓ Check the matrix QC sample that it meets the acceptance criteria or note possible matrix interference in the run log.
- ✓ Check that all samples are analyzed within the calibration range. Dilute the samples that measured over the absorbance of the highest calibration point.
- ✓ Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Check the calibration checks to make sure that the instrument is functioning properly.

10.4.8.2. Refer to Sec. 11 for quality control and Sec. 12 for corrective action.

10.4.8.3. Refer to Appendix 1 for acceptance criteria. If any of the acceptance criteria is not met, perform the necessary corrective action. If problem persists, inform the Supervisor for further action.

10.4.8.4. Properly fill up the analytical run log.

10.4.8.5. Upload the electronic data to the network.

10.4.9. Dealing with Carryover

10.4.9.1. Check the sample analyzed preceded by another sample found to have target analyte concentrations exceeding the calibration range.

10.4.9.2. If the target analyte is not detected, as found in the preceding high concentration sample, proceed with data reduction.

10.4.9.3. If the target analyte is detected, as found in the 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.

10.4.10. Method of Standard Addition (MSA)

10.4.10.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.10.2. 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)

Ms 3 – take 10 ml of digestate and add 0.6 ml of 100 µg/L spike standard (≈ 8 µg/L)

10.4.10.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq.-10.5.5.

10.5. **Calculations**

10.5.1. Calibration Factor

Plot the absorbance (y-axis) versus the known concentration of the calibration standards (x-axis) using Excel spreadsheet or equivalent. The reciprocal of the slope shall be the calibration factor.

10.5.2. Calculate for Mercury Concentration

$$C = [(A + CF) - Y] * DF \qquad \text{Eq.-10.5.2}$$

where:

C	–	Concentration of Mercury, µg/L
A	–	Absorbance
CF	–	Calibration Factor = 1/slope
Y	–	Y-intercept / slope
DF	–	Dilution Factor

10.5.3. Calculate for Percent Recovery

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.5.3}$$

where:

C_f	–	Concentration found
C	–	Concentration of the sample (use 0 for LCS)
C_s	–	Concentration of spike

10.5.4. Calculate for Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.4}$$

where:

RPD	–	Relative Percent Difference
C_1	–	Measured concentration of the first sample aliquot
C_2	–	Measured concentration of the second sample aliquot

10.5.5. Calculation for MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \qquad \text{Eq.-10.5.5}$$

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 water

10.6. **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.

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 and sample results using HGF1.exe.

10.7.2. Generate the QC summaries using HGF3.exe.

10.7.3. Generate the case narrative using CN00.exe.

10.7.4. Arrange the analysis package in sequence as detailed below.

10.7.4.1. Case Narrative

10.7.4.2. Lab Chronicle

10.7.4.3. Sample Results

10.7.4.4. LCS/LCSD Summary

10.7.4.5. MS/MSD Summary

10.7.4.6. Post Digestion Spike Summary

10.7.4.7. Dilution Test Result Summary

10.7.4.8. Analytical Run Log

10.7.4.9. Raw Data

10.7.4.10. Sample Preparation Log

10.7.4.11. Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check method blank is compliant to Project Specific Requirements (PSR) criteria.
- ✓ 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.

10.8.2. 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% $SnCl_2$, troubleshooting and major repairs must be recorded in the maintenance log. Refer to Form 7470FM.

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. Pipettes must be calibrated prior to its use. Refer to EMAX-QC06.

11.1.2. Reagents are subjected to QC check prior to its use. Refer to EMAX-QC01.

11.1.3. Properly treat all lab wares used in the sample preparations as specified in EMAX-QC07.

11.1.4. 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.

11.1.5. 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.

11.2. **Sample Analysis QC**

11.2.1. Every analytical run is preceded by an Initial Calibration (ICAL) and 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 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.

11.2.3. Evaluate MS/MSD to document matrix interference.

11.2.4. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.5. Perform Post Digestion Spike result if the recoveries of MS/MSD fail and if dilution test failed.

11.2.6. Use Method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.7. Refer to Appendix 1 for acceptance criteria.

11.3. **Method QC**

11.3.1. Detection Limit study must be established before the analytical procedure can be used. Quarterly verification must be performed. Refer to EMAX-QA04.

11.3.2. 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 $SnCl_2$. 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 Sec. 12.1.1 prior to re-calibration.

12.2.3. Continuing Calibration Verification (CCV) - If CCV is non-compliant, consider the following suggestion to correct the problem:

- 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 Sec. 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 suggestion 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 / Matrix Spike Duplicate (MS/MSD) - 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.

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^{-3} 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 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.

13.4. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.5. To prevent internal pollution, the entire sample preparation process and analytical standard preparation must be conducted in a properly functioning fume hood.

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%.

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. Safety Data Sheet (SDS) – 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) and Department of Energy (DOE) 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 ELAP Quality Systems Manual (QSM), 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

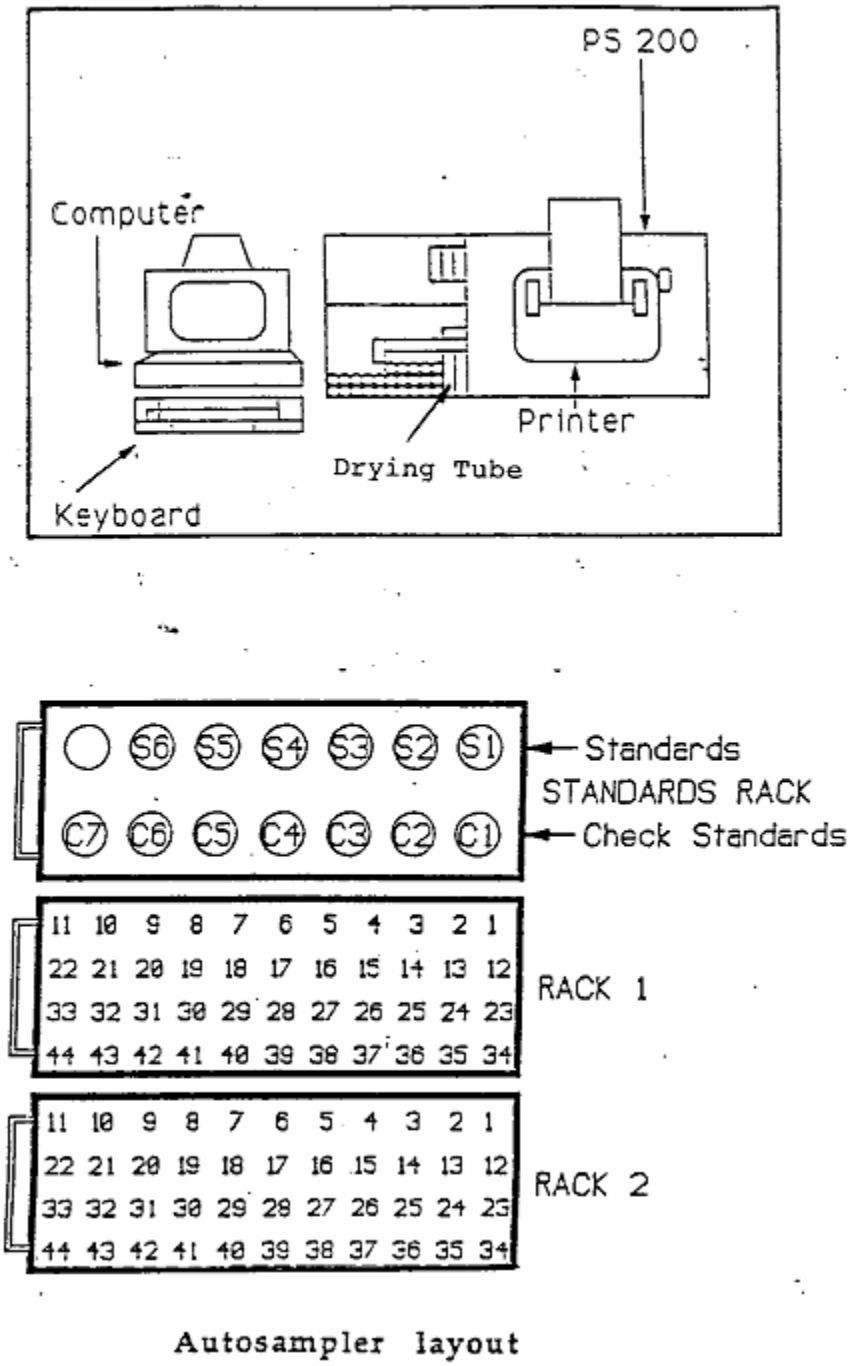
17.3. **Forms**

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

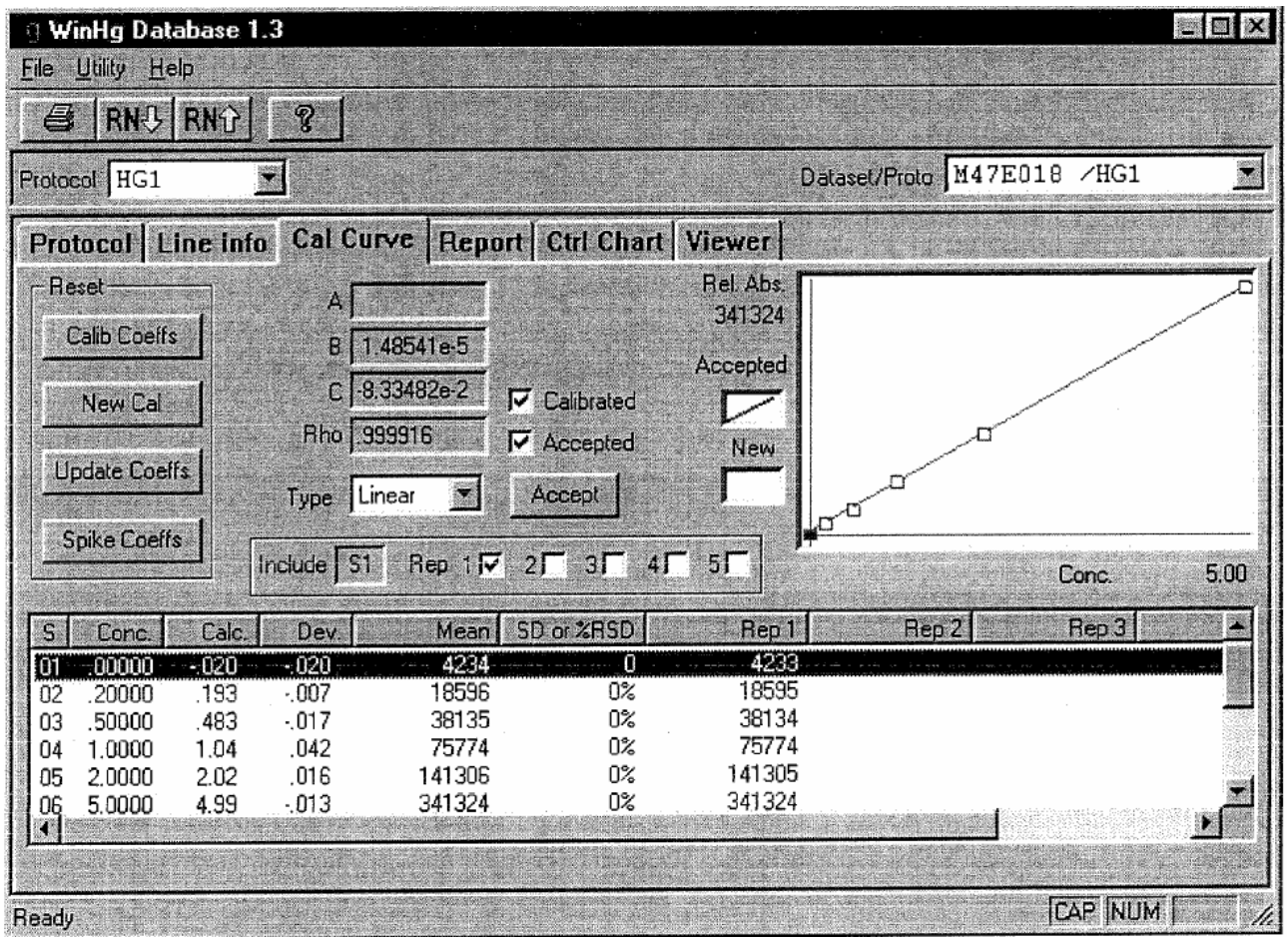


Figure 3: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD SW7470A
MERCURY BY COLD VAPOR

Client : XYZ, INC.
Project : CLEAN WATER
Batch No. : 16E175
Matrix : WATER
InstrumentID : 47

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (ug/L)	DIL'N FACTOR	MOIST (%)	LOQ (ug/L)	DL (ug/L)	LOD (ug/L)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
MBLK1W	HGE027WB	ND	1	NA	0.50	0.054	0.10	05/31/1620:45	05/31/1614:00	M47E018043	M47E018	HGE027W	NA	NA
LCS1W	HGE027WL	2.48	1	NA	0.50	0.054	0.10	05/31/1620:47	05/31/1614:00	M47E018044	M47E018	HGE027W	NA	NA
LCD1W	HGE027WC	2.46	1	NA	0.50	0.054	0.10	05/31/1620:54	05/31/1614:00	M47E018047	M47E018	HGE027W	NA	NA
03M08-2016S	E175-08	ND	1	NA	0.50	0.054	0.10	05/31/1620:58	05/31/1614:00	M47E018049	M47E018	HGE027W	05/18/1614:28	05/20/16
03M08-2016SMS	E175-08M	2.59	1	NA	0.50	0.054	0.10	05/31/1621:03	05/31/1614:00	M47E018051	M47E018	HGE027W	05/18/1614:28	05/20/16
03M08-2016SMSD	E175-08S	2.59	1	NA	0.50	0.054	0.10	05/31/1621:05	05/31/1614:00	M47E018052	M47E018	HGE027W	05/18/1614:28	05/20/16
03M12B-2016S	E175-01	ND	1	NA	0.50	0.054	0.10	05/31/1621:07	05/31/1614:00	M47E018053	M47E018	HGE027W	05/18/1611:16	05/20/16
03M12B-2016S-9	E175-02	ND	1	NA	0.50	0.054	0.10	05/31/1621:10	05/31/1614:00	M47E018054	M47E018	HGE027W	05/18/1611:16	05/20/16
03M04B-2016S	E175-03	ND	1	NA	0.50	0.054	0.10	05/31/1621:12	05/31/1614:00	M47E018055	M47E018	HGE027W	05/18/1612:56	05/20/16
03M11B-2016S	E175-04	ND	1	NA	0.50	0.054	0.10	05/31/1621:14	05/31/1614:00	M47E018056	M47E018	HGE027W	05/18/1614:42	05/20/16
03M10-2016S	E175-05	ND	1	NA	0.50	0.054	0.10	05/31/1621:21	05/31/1614:00	M47E018059	M47E018	HGE027W	05/18/1616:22	05/20/16
03MW20-2016S	E175-06	ND	1	NA	0.50	0.054	0.10	05/31/1621:23	05/31/1614:00	M47E018060	M47E018	HGE027W	05/18/1611:28	05/20/16
03M18-2016S	E175-07	ND	1	NA	0.50	0.054	0.10	05/31/1621:25	05/31/1614:00	M47E018061	M47E018	HGE027W	05/18/1611:35	05/20/16
03M19-2016S	E175-09	ND	1	NA	0.50	0.054	0.10	05/31/1621:27	05/31/1614:00	M47E018062	M47E018	HGE027W	05/19/1611:17	05/20/16

Figure 4: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LAB CONTROL SAMPLE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX	WATER		% MOISTURE: N/A
DILUTION FACTOR	1	1	1
SAMPLE ID	MBLK1W	LCS1W	LCD1W
LAB SAMPLE ID	HGE027WB	HGE027WL	HGE027WC
LAB FILE ID	M47E018043	M47E018044	M47E018047
DATE PREPARED	05/31/1614:00	05/31/1614:00	05/31/1614:00
DATE ANALYZED	05/31/1620:45	05/31/1620:47	05/31/1620:54
PREP BATCH	HGE027W	HGE027W	HGE027W
CALIBRATION REF	M47E018	M47E018	M47E018

ACCESSION:

PARAMETER	MB RESULT (ug/L)	SPIKE AMT (ug/L)	BS RESULT (ug/L)	BS REC (%)	SPIKE AMT (ug/L)	BSD RESULT (ug/L)	BSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	2.50	2.48	99	2.50	2.46	98	1	80-120	20

Figure 5: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX :	WATER		% MOISTURE: NA
DILUTION FACTOR:	1	1	1
SAMPLE ID :	03M08-2016S	03M08-2016SMS	03M08-2016SMSD
LAB SAMPLE ID :	E175-08	E175-08M	E175-08S
LAB FILE ID :	M47E018049	M47E018051	M47E018052
DATE PREPARED :	05/31/1614:00	05/31/1614:00	05/31/1614:00
DATE ANALYZED :	05/31/1620:58	05/31/1621:03	05/31/1621:05
PREP BATCH :	HGE027W	HGE027W	HGE027W
CALIBRATION REF:	M47E018	M47E018	M47E018

ACCESSION:

PARAMETER	PARENT RESULT (ug/L)	SPIKE AMT (ug/L)	MS RESULT (ug/L)	MS REC (%)	SPIKE AMT (ug/L)	MSD RESULT (ug/L)	MSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	2.50	2.59	104	2.50	2.59	104	0	80-120	20

Figure 6: TYPICAL ANALYTICAL SPIKE SUMMARY

EMAX QUALITY CONTROL DATA
ANALYTICAL SPIKE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

MATRIX : WATER % MOISTURE: NA

DILUTION FACTOR:	1	1
SAMPLE ID :	03M08-2016S	03M08-2016SMS
LAB SAMPLE ID :	E175-08	E175-08A
LAB FILE ID :	M47E018049	M47E018048
DATE PREPARED :	05/31/1614:00	05/31/1614:00
DATE ANALYZED :	05/31/1620:58	05/31/1621:03
PREP BATCH :	HGE027W	HGE027W
CALIBRATION REF:	M47E018	M47E018

ACCESSION:

PARAMETER	SMPL RESULT (ug/L)	SPIKE AMT (ug/L)	AS RSLT (ug/L)	MS REC (%)	QC LIMIT (%)
Mercury	ND	2.50	2.58	103	85-115

Figure 7: TYPICAL DILUTION TEST SUMMARY

EMAX QUALITY CONTROL DATA
SERIAL DILUTION ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN WATER
BATCH NO. : 16E175
METHOD : SW7470A

==========

MATRIX : WATER % MOISTURE: NA

DILUTION FACTOR:	1	1
SAMPLE ID :	03M08-2016S	03M08-2016SMS
LAB SAMPLE ID :	E175-08	E175-08J
LAB FILE ID :	M47E018049	M47E018050
DATE PREPARED :	05/31/1614:00	05/31/1614:00
DATE ANALYZED :	05/31/1620:58	05/31/1621:01
PREP BATCH :	HGE027W	HGE027W
CALIBRATION REF:	M47E018	M47E018

ACCESSION:

PARAMETER	SMPL RESULT (ug/L)	SERIAL DIL RSLT (ug/L)	DIL RST (%)	QC LIMIT (%)
Mercury	ND	ND	0	10

Figure 8: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ,INC.

Project: CLEAN WATER

SDG : 16E175

METHOD SW7470A
MERCURY BY COLD VAPOR

A total of nine (9) water samples were received on 05/20/16 to be analyzed for Mercury by Cold Vapor in accordance with Method SW7470A and project specific requirements.

Holding Time
Samples were digested and 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.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed. Mercury was not detected in HGE027WB. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. HGE027WL/HGE027WC was within LCS limits. Refer to LCS summary form for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed. Mercury was within MS QC limits in E175-08M/S. Refer to Matrix QC summary form for details. 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. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURES

PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Initial multipoint calibration	Daily initial calibration prior to sample analysis	Correlation coefficient r≥ 0.995 for linear regression	Correct the problem then repeat initial calibration		
Initial calibration verification (second source)	Daily after initial calibration	Analyte within ± 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 ± 10% 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 > 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	%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≤ 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 (~5x the LOQ after dilution), otherwise perform MSA.		
Dilution Test	When Post-Digestion Spike fails and analyte concentration is sufficiently high (~5x the LOQ after dilution)	Within ± 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
EPA 7470A

EMAX-7470 Rev. 7
Conc Unit: mg/L
Sample Amount(mL): 50

Extraction dates: 1/11/2016
Analysis dates: 1/11/2016
Extracted and Analyzed by: N. Tan

PARAMETER	HGA006WL	HGA006WC	HGA007WL	HGA007WC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	M47A004012	M47A004013	M47A004043	M47A004044							
Mercury	2.56	2.56	2.47	2.51	2.5	2.53	101	0.044	2	80 - 120	PASSED

7470FS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

DIGESTION LOG *for* MERCURY

Page 1

Note: For samples, relevant QCs/Standards digested, refer to attached digestion sequence.

Comments:

Digestion Vessel Lot #:

Book #: E47-102

Batch No.:

Matrix:

SOP #	Rev. #
☐ EMAX-7470	8
☐ EMAX-7471	9
☐ EMAX-245.1	4
☐ EMAX-	

Standards	ID	Conc. (µg/L)	Amount Added (ml)
ICAL			
CCV			
ICV			
LCS/MS			

Reagent	ID / Lot #
HNO_3	
HCl	
H_2SO_4	
$KMnO_4$	
$K_2S_2O_8$	
$NH_2OH{\bullet}HCl{\bullet}NaCl$	
$SnCl_2$	
Silica Sand	
Reagent Water	
pH strip 0-14	

Digestor ID/ Temp (°C)

Thermometer ID/LOC:

Thermometer ID/LOC:

Pipette ID:

☐ H_2SO_4 dispenser checked @ 2.5 ml with Class A graduated cylinder

☐ HCl dispenser checked @ ___ ml with Class A graduated cylinder

☐ HNO_3 dispenser checked @ ___ ml with Class A graduated cylinder

Prepared By:

Standard Added By: Witnessed By:

7470FA: ANALYTICAL RUN LOG

EMAX LABORATORIES INC

ANALYSIS RUN LOG
for
MERCURY

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Start Date: Start Time:

End Date: End Time:

Comments:

Book #: A47-085

Instrument No.: 47

Analytical Sequence/Batch:

Method File:

Micropipette ID: ☐ ICP DOE-1

Micropipette ID: ☐ HG-03

Micropipette ID: ☐ HG-04

Micropipette ID: ☐

SOP #	Rev. #
☐ EMAX-7470	
☐ EMAX-7471	
☐ EMAX-245.1	
☐ EMAX-	

STANDARDS ID
S1
S2
S3
S4
S5
S6
CCV
ICV
LCS
Analytical Spike

Analyzed By:

Date

7470FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 47 Month: Year: 201_ Book #: M47-010

DAQ TEST							
Sample: 200/0.01 = 1/20,000							
Hg Lamp Volt: 4.0/0.5							
GAS CONTROL TEST							
Argon Input Pressure: 75-80 psi							
Power Supply Voltage: 5V+/-0.25, +12V+/-0.5, -12V+/-0.5							
Gas: 0-1.0 LPM							
Pump Rate: 2-15 ml/min							
Instrument Warm-up: Minimum 5 mins							
Peristaltic Pump							
Tubings Pump/Capillary							
Fume Hood							
Reductant Bottle							
Rinse Solution							
Computer Date/Time							
Printer Paper							
Analyst Initial							
Trouble Shooting							
Problem(s) Encountered							
Return to Specs (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

STANDARD OPERATING PROCEDURE

MERCURY IN SOLID OR SEMISOLID WASTE

SOP No.:	EMAX-7471	Revision No.	9	Effective Date:	13-Jul-16
Prepared By:	Mary Jane Mendoza			Date:	07.12.16
Approved By:	Kenette Pimentel QA Manager			Date:	07.12.16
Approved By:	Caspar Pang Laboratory Director			Date:	07-12-16

Control Number: 7471-09-

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

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 Preservation**

5.1.1. Samples are expected to be contained in a jar, Shelby tube or as specified by the project. cooled to ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. Digest all samples within 28 days from date of collection.

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-QC04 Balance Calibration

6.8. EMAX-QC06 Volumetric Labwares and Micropipette Verification

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, 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 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, Win Hg Runner 1.3.

8.1.1.2. Leeman Hydra AA Automated Analyzer with Autosampler, Computer, Printer and PS200 Software, Win Hg Runner 1.3.

8.1.2. 100 ml Digestion vessel

8.1.3. Digestion block or equivalent.

8.1.4. Micropipettes and tips

8.1.5. Thermometer

8.2. **Chemicals and Reagents**

8.2.1. Where available, purchase reagent grade chemicals and reagents

8.2.2. Hydrochloric acid, concentrated.

8.2.3. Nitric acid, concentrated

8.2.4. Stannous Chloride: Dissolve 200 g. of $SnCl_2$ in reagent water, add 200 ml concentrated HCl and dilute to 2 L.

8.2.5. Sodium chloride – hydroxylamine hydrochloride solution. Dissolve 120g of sodium chloride and 120 g of hydroxylamine hydrochloride in reagent water and dilute to 1 L. *(Note:Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.)*

8.2.6. Potassium permanganate, 5% solution. Dissolve 50 g potassium permanganate in 1 L reagent water.

8.2.7. Agua Regia – 1 part HNO_3 : 3 parts HCl solution

8.2.8. Reagent water – mercury-free water

8.2.9. Silica sand for blank soil matrix

1.0 STANDARDS

9.1. Refer to EMAX-QC02 for proper analytical standard preparation.

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.

Stock Std	Name	Source	Conc.	Used for
Primary	Mercury	ERA or equivalent	1000 mg/L	ICAL, CCV
Secondary	Mercury	AccuStandard or equivalent	1000 mg/L	ICV/LCS/MS

9.3.2. Transfer standards on a properly labeled inert vial with minimal headspace and store at -10°C to -20°C.

9.4. **Intermediate Standard Solution**

9.4.1. Primary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg primary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.2. Secondary Intermediate Standard, 10.0 mg/L: Dilute 1.0 mL of 1000 mg/L Hg secondary stock standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.4.3. Post Spike Standard, 100 µg/L: Dilute 1.0 mL of 10 mg/L Hg primary intermediate standard to 100 mL using reagent water. Digest prior to use.

9.5. **Working Standard**

9.5.1. Primary Working Standard, 100 µg/L: Dilute 1.0 mL of 10 mg/L primary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.5.2. Secondary Working Standard, 100 µg/L: Dilute 1.0 mL of 10 mg/L secondary intermediate standard to 100 mL using reagent water. Transfer the standard in a clean and properly labeled container.

9.6. **Initial Calibration Standards (ICAL)**

9.6.1. Initial calibration consists of five standards and a reagent water blank. Using the primary working standard, prepare the ICAL points as follows:.

Level	Primary Working Standard, 100 µg/L Aliquot (mL)	Concentration (µg/L)
S0	0	0
S1	0.2	0.2
S2	0.5	0.5
S3	1.0	1.0
S4	2.0	2.0
S5	5.0	5.0

Note: Other concentration levels may be prepared as appropriate to meet project quality objectives.

9.7. **Initial Calibration Verification Standard (ICV)**

9.7.1. Prepare ICV at 2 µg/L with 2mL of secondary working standard (100 µg/L) in a digestion vessel.

9.8. **Continuing Calibration Verification Standard (CCV)**

9.8.1. Prepare CCV at 2 µg/L with 2 mL of primary working standard (100 µg/L) in a digestion vessel.

9.9. **Laboratory Control Standard (LCS) / Matrix Spike (MS) Standard**

9.9.1. Spike 2.5mL of secondary working standard (100 µg/L) to 0.6g soil blank (for LCS) or designated sample (for MS).

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Weigh a representative 0.6 g portion of sample into digestion vessels.

10.1.2. Add spike standards to the LCS/MS (Sec. 9.9.1). Add appropriate standards for Initial Calibration (Sec. 9.6.1), ICV (Sec. 9.7.1) and CCVs (Sec. 9.8.1) in clean digestion vessels.

10.1.2.1. **Spike Addition.** Call a witness. Have the witness verify the setting of the micropipette and the expiration date of the spike standard.

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°C ± 3°C in the digestion block, cool and add 50 mL of reagent water.

10.1.5. Add 15 ml of 5% $KMnO_4$ 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. The maximum amount of permanganate that can be added is 25 mL.

Add the same amount of permanganate solution added 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 ± 3°C.

10.1.8. Allow the samples to cool.

10.1.9. Add 6 ml NaCl-hydroxylamine hydrochloride solution and wait for sample to decolorize. Additional NaCl-hydroxylamine hydrochloride may be added to reduce excess permanganate in 1 mL increments up to maximum total amount of 10 mL.

Add the same amount of NaCl-hydroxylamine hypochloride solution added to the method blank, LCS, calibration standards and calibration verification standards.

10.1.10. Upon decolorization, dilute to 100 mL using reagent water.

10.1.11. 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
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	50 sec.	60 sec.
CALIBRATION S0,S1, S2, S3, S4, S5	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0	Concentration, µg/L 0 0,.20, 0.50, 1.0, 2.0, 5.0

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 Sec. 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.

10.3.1.4. Check that the reductant solution, 10% $SnCl_2$, 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 standards as described in Sec.9.6.1.

10.3.2.2. Digest the ICAL standards as described in Sec. 10.1.

10.3.2.3. Analyze as described in Sec. 10.4.

10.3.2.4. Refer to Sec. 10.5 for calculations.

10.3.2.5. Initiate initial calibration as described in the instrument operations manual. After calibration is completed, acquire the calibration data and review.

10.3.2.6. Refer to Appendix 1 for acceptance criteria.

10.3.2.7. Verify the initial calibration by a secondary source standard (ICV).

10.3.3. Initial Calibration Verification (ICV)

10.3.3.1. Prepare ICV as described in Sec. 9.7.1. Prepare ICB using reagent water.

10.3.3.2. Digest the ICV and ICB as described in Sec. 10.1.

10.3.3.3. Analyze the ICV to verify the concentration of the ICAL. Analyze the ICB after the ICV.

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 Sec. 9.8.1. Prepare CCB using reagent water.

10.3.4.2. Digest the CCV and CCB as described in Sec. 10.1.

10.3.4.3. Analyze the CCV to verify the validity of the ICAL. Analyze the CCB after the CCV.

10.3.4.4. Refer to Appendix 1 for 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**

S0	0.00000
S1	0.20000
S2	0.50000
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 Digestion Spike (Sec. 10.4.4)

10.4.2.9. Parent Sample

10.4.2.10. Serial Dilution (Sec. 10.4.5)

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. Using the analytical sequence, arrange the digested standards and samples to be analyzed chronologically.

10.4.4. 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.

10.4.5. Prepare a Dilution Test sample at 5x dilution. Pipette 2 mL of digestate, add 8 mL of S0 into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.

10.4.6. Prepare a Post Digestion Spike test sample. Using 10 mL unspiked sample digestate (preferably the QC sample), add 0.3 mL Post Spike standard (Sec. 9.4.3).

10.4.7. Set the prepared samples into the autosampler and start the analytical run.

10.4.8. Sample Result Evaluation

10.4.8.1. Check the QC data as soon as available.

- ✓ Check the ICAL and ICV prior to sample analysis.
- ✓ Check the method blank and LCS against project specific requirements.
- ✓ Check the matrix QC sample that it meets the acceptance criteria or note possible matrix interference in the run log.
- ✓ Check that all samples are analyzed within the calibration range. Dilute samples that measured over the absorbance of the highest calibration point.
- ✓ Check that the analytical data generated indicating positive results are qualitatively and quantitatively correct.
- ✓ Check the calibration checks to make sure that the instrument is functioning properly.

10.4.8.2. Refer to Sec. 11 for quality control and Sec. 12 for corrective action.

10.4.8.3. Refer to Appendix 1 for acceptance criteria. If any of the acceptance criteria is not met, perform the necessary corrective action. If problem persists, inform the Supervisor for further action.

10.4.8.4. Properly fill up the analytical run log.

10.4.8.5. Upload the electronic data to the network.

10.4.9. Dealing with Carryover

10.4.9.1. Check the sample analyzed preceded by another sample found to have target analyte concentrations exceeding the calibration range.

10.4.9.2. If no target analyte is detected as found in preceding high concentration sample, proceed with data reduction.

10.4.9.3. 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.

10.4.10. Method of Standard Addition (MSA)

10.4.10.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.10.2. 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)

10.4.10.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq.-10.5.5.

10.5. **Calculations**

10.5.1. Calibration Factor

Plot the absorbance (y-axis) versus the known concentration of the calibration standards (x-axis) using Excel spreadsheet or equivalent. The reciprocal of the slope shall be the calibration factor.

10.5.2. Calculate for Mercury Concentration

$$C = [(A + CF) - Y] * DF * Vd / (S * 1000)$$ Eq.-10.5.2

where:

C	–	Concentration of Mercury, mg/Kg
A	–	Absorbance
CF	–	Calibration Factor = 1/slope
Y	–	Y-intercept / slope
DF	–	Dilution Factor
Vd	–	Final Digestate Volume, mL
S	–	Sample amount, g

10.5.3. Calculate for Percent Recovery

$$\%\,\mathrm{Recovery} = \frac{(Cf - C)}{Cs} x100$$ Eq.-10.5.3

where:

Cf	–	Found Concentration
C	–	Sample Concentration (for LCS, C=0)
Cs	–	Spike Concentration

10.5.4. Calculate for RPD

$$RPD = \frac{|C1 - C2|}{\left(\frac{C1 + C2}{2}\right)} x100$$ Eq.-10.5.4

where:

RPD	–	Relative Percent Difference
$C1$	–	Measured concentration of the first sample aliquot
$C2$	–	Measured concentration of the second sample aliquot

10.5.5. Calculation for MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x}$$ Eq.-10.5.5

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 water

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 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 and sample results using HGF1.exe.

10.7.2. Generate the QC summaries using HGF3.exe.

10.7.3. Generate the case narrative using CN00.exe.

10.7.4. Arrange the analysis package in sequence as detailed below:

10.7.4.1. Case Narrative

10.7.4.2. Lab Chronicle

10.7.4.3. Sample Results

10.7.4.4. LCS/LCSD Summary

10.7.4.5. MS/MSD Summary

10.7.4.6. Post Digestion Spike Summary

10.7.4.7. Dilution Test Result Summary

10.7.4.8. Analytical Run Log

10.7.4.9. Raw Data

10.7.4.10. Sample Preparation Log

10.7.4.11. Non-Conformance Report (if any)

10.8. **Dsta Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Check that the method blank is compliant to Project Specific Requirement (PSR).

- ✓ Check LCS/LCSD, MS/MSD and dilution test against 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 that these are properly filled.
- ✓ 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.

10.8.2. 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 including checking of reductant solution (10% $SnCl_2$), troubleshooting and major repairs in the maintenance log. Refer to Form 7471FM.

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. Pipettes must be calibrated daily prior to use. Refer to EMAX-QC06.

11.1.2. Reagents are subject to QC check prior to use. Refer to EMAX-QC01.

11.1.3. Properly treat all labwares used in sample preparation as specified in EMAX-QC07.

11.1.4. 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.

11.1.5. 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.

11.2. **Sample Analysis QC**

11.2.1. Every analytical run is 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 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.

11.2.3. Evaluate MS/MSD to document matrix interference.

11.2.4. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.5. Perform Post Digestion Spike whenever recoveries of MS/MSD fail and if the dilution test failed.

11.2.6. Use Method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.7. Refer to Appendix 1 for acceptance criteria.

11.3. **Method QC**

11.3.1. Detection Limit study must be established before the analytical procedure can be used. Quarterly verification must be performed. Refer to EMAX-QA04.

11.3.2. All analysts conducting this analysis must have established Demonstration of Capability (DOC) as described in EMAX-QA05.

12.0 CORRECTIVE ACTION

12.1. Corrective action for each Quality Control Procedure is summarized in Appendix 1.

12.2. **Calibration**

12.2.1. Initial Calibration (ICAL) - 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 $SnCl_2$. 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.

12.2.2. Initial Calibration Verification (ICV) - If the ICV is non-compliant, consider the following suggestion 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 Sec. 12.2.1 prior to re-calibration.

12.2.3. Continuing Calibration Verification (CCV) - If the CCV is non-compliant, consider the following suggestion 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 Sec. 12.2.1 prior to re-calibration.

12.3. **Sample Preparation**

12.3.1. Method Blank (MB) - If method blank is non-compliant, consider the following suggestion to correct the problem:

- Check the sample results. If sample results are non-detect, you may report the result upon concurring with the PM. Otherwise, perform the corrective action as specified in the PSR.

12.4. **Sample Analysis**

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 reanalyze 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. Redigest to include the associated sample(s).

12.4.2. Matrix Spike / Matrix Spike Duplicate (MS/MSD) - 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 $\leq$ 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.

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.

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^{-3} 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.

13.4. Observe all necessary precautions to avoid spillage of solvent that may go to wastewater drains.

13.5. To prevent internal pollution, the entire sample preparation process and analytical standard preparation must be conducted in a properly functioning fume hood.

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 "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. 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.

15.2.8. 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 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) and Department of Energy (DOE) Projects**

15.4.1. 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. 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.

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 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

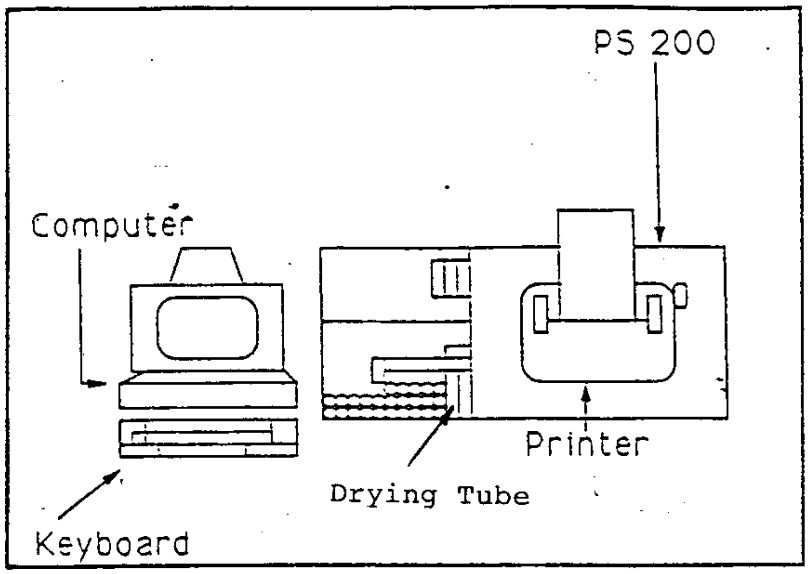
17.3. **Forms**

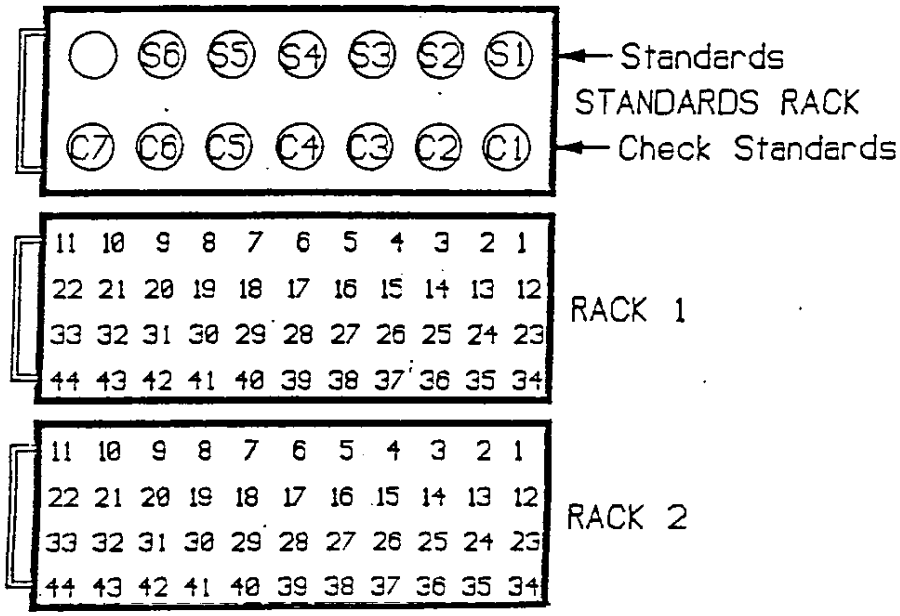
17.3.1. 7471FS Sample Preparation Log

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

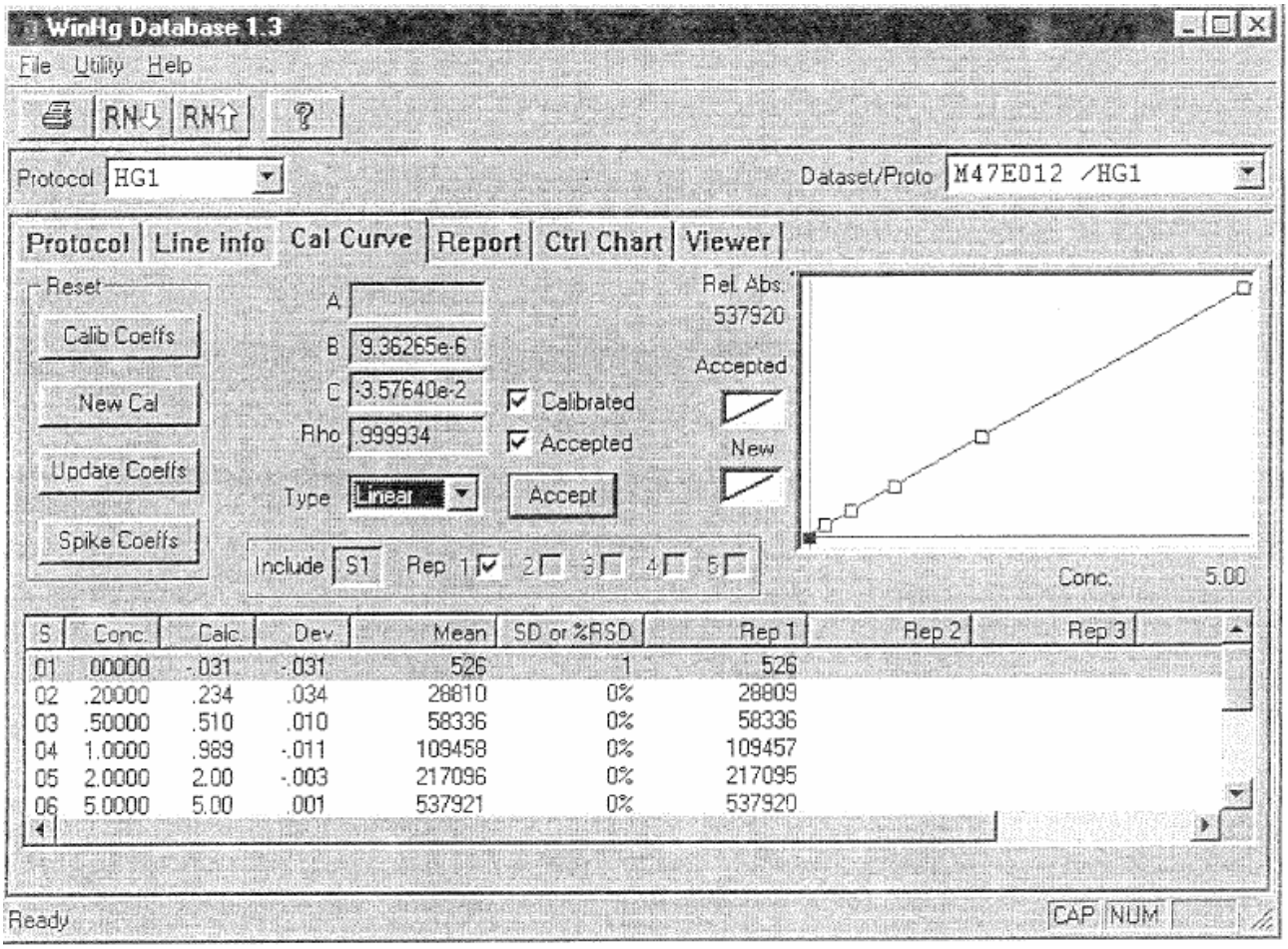


Figure 3: **TYPICAL SAMPLE RESULT SUMMARY**

METHOD SW7471B
MERCURY BY COLD VAPOR

Client : XYZ, INC.
Project : CLEAN LAND PROJECT
Batch No. : 16E157
Matrix : SOIL
InstrumentID : 47

CLIENT SAMPLE ID	EMAX SAMPLE ID	RESULTS (mg/kg)	DIL'N FACTOR	MOIST (%)	LOQ (mg/kg)	DL (mg/kg)	LOD (mg/kg)	ANALYSIS DATETIME	PREPARATION DATETIME	DATA FILE ID	CAL REF	PREP BATCH	COLLECTION DATETIME	RECEIVED DATETIME
MBLK1S	HGE015SB	ND	1	NA	0.10	0.010	0.020	05/20/1616:17	05/20/1612:00	M47E012011	M47E012	HGE015S	NA	NA
LCS1S	HGE015SL	0.419	1	NA	0.10	0.010	0.020	05/20/1616:19	05/20/1612:00	M47E012012	M47E012	HGE015S	NA	NA
LCD1S	HGE015SC	0.415	1	NA	0.10	0.010	0.020	05/20/1616:21	05/20/1612:00	M47E012013	M47E012	HGE015S	NA	NA
A	E157-01	ND	1	15.0	0.12	0.012	0.023	05/20/1616:26	05/20/1612:00	M47E012015	M47E012	HGE015S	05/16/1610:00	05/18/16
AMS	E157-01M	0.515	1	15.0	0.12	0.012	0.023	05/20/1616:30	05/20/1612:00	M47E012017	M47E012	HGE015S	05/16/1610:00	05/18/16
AMSD	E157-01S	0.508	1	15.0	0.12	0.012	0.023	05/20/1616:32	05/20/1612:00	M47E012018	M47E012	HGE015S	05/16/1610:00	05/18/16
B	E157-02	ND	1	12.5	0.11	0.011	0.023	05/20/1616:35	05/20/1612:00	M47E012019	M47E012	HGE015S	05/16/1610:15	05/18/16
C	E157-03	ND	1	15.8	0.12	0.012	0.023	05/20/1616:37	05/20/1612:00	M47E012020	M47E012	HGE015S	05/16/1610:30	05/18/16
D	E157-04	ND	1	11.2	0.11	0.011	0.022	05/20/1616:44	05/20/1612:00	M47E012023	M47E012	HGE015S	05/16/1610:45	05/18/16
E	E157-05	0.0204J	1	17.5	0.12	0.012	0.024	05/20/1616:46	05/20/1612:00	M47E012024	M47E012	HGE015S	05/16/1611:15	05/18/16

Figure 4: **TYPICAL LCS/LCSD SUMMARY**

EMAX QUALITY CONTROL DATA
LAB CONTROL SAMPLE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN LAND PROJECT
BATCH NO. : 16E157
METHOD : SW7471B

MATRIX	SOIL		% MOISTURE: N/A
DILUTION FACTOR	1	1	1
SAMPLE ID	MBLK1S	LCS1S	LCD1S
LAB SAMPLE ID	HGE015SB	HGE015SL	HGE015SC
LAB FILE ID	M47E012011	M47E012012	M47E012013
DATE PREPARED	05/20/1612:00	05/20/1612:00	05/20/1612:00
DATE ANALYZED	05/20/1616:17	05/20/1616:19	05/20/1616:21
PREP BATCH	HGE015S	HGE015S	HGE015S
CALIBRATION REF	M47E012	M47E012	M47E012

ACCESSION:

PARAMETER	MB RESULT (mg/kg)	SPIKE AMT (mg/kg)	BS RESULT (mg/kg)	BS REC (%)	SPIKE AMT (mg/kg)	BSD RESULT (mg/kg)	BSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	0.416	0.419	101	0.415	0.415	100	1	80-120	20

Figure 5: **TYPICAL MS/MSD SUMMARY**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN LAND PROJECT
BATCH NO. : 16E157
METHOD : SW7471B

MATRIX	SOIL		% MOISTURE: 15.0
DILUTION FACTOR	1	1	1
SAMPLE ID	A	AMS	AMSD
LAB SAMPLE ID	E157-01	E157-01M	E157-01S
LAB FILE ID	M47E012015	M47E012017	M47E012018
DATE PREPARED	05/20/1612:00	05/20/1612:00	05/20/1612:00
DATE ANALYZED	05/20/1616:26	05/20/1616:30	05/20/1616:32
PREP BATCH	HGE015S	HGE015S	HGE015S
CALIBRATION REF	M47E012	M47E012	M47E012

ACCESSION:

PARAMETER	PARENT RESULT (mg/kg)	SPIKE AMT (mg/kg)	MS RESULT (mg/kg)	MS REC (%)	SPIKE AMT (mg/kg)	MSD RESULT (mg/kg)	MSD REC (%)	RPD (%)	QC LIMIT (%)	MAX RPD (%)
Mercury	ND	0.488	0.515	106	0.483	0.508	105	1	80-120	20

Figure 6: TYPICAL DILUTION TEST SUMMARY

EMAX QUALITY CONTROL DATA
SERIAL DILUTION ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN LAND PROJECT
BATCH NO. : 16E157
METHOD : SW7471B

MATRIX : SOIL % MOISTURE: 15.0

DILUTION FACTOR:	1	1
SAMPLE ID :	A	AMS
LAB SAMPLE ID :	E157-01	E157-01J
LAB FILE ID :	M47E012015	M47E012016
DATE PREPARED :	05/20/1612:00	05/20/1612:00
DATE ANALYZED :	05/20/1616:26	05/20/1616:28
PREP BATCH :	HGE015S	HGE015S
CALIBRATION REF:	M47E012	M47E012

ACCESSION:

PARAMETER	SMPL RESULT (mg/kg)	SERIAL DIL RSLT (mg/kg)	DIF RSLT (%)	QC LIMIT (%)
Mercury	ND	ND	0	10

Figure 7: TYPICAL POST DIGESTION SPIKE SUMMARY

EMAX QUALITY CONTROL DATA
ANALYTICAL SPIKE ANALYSIS

CLIENT : XYZ, INC.
PROJECT : CLEAN LAND PROJECT
BATCH NO. : 16E157
METHOD : SW7471B

MATRIX : SOIL % MOISTURE: 15.0

DILUTION FACTOR:	1	1
SAMPLE ID :	A	AMS
LAB SAMPLE ID :	E157-01	E157-01A
LAB FILE ID :	M47E012015	M47E012014
DATE PREPARED :	05/20/1612:00	05/20/1612:00
DATE ANALYZED :	05/20/1616:26	05/20/1616:26
PREP BATCH :	HGE015S	HGE015S
CALIBRATION REF:	M47E012	M47E012

ACCESSION:

PARAMETER	SMPL RESULT (mg/kg)	SPIKE AMT (mg/kg)	AS RSLT (mg/kg)	AS % REC	QC LIMIT (%)
Mercury	ND	0.488	0.508	104	85-115

Figure 8: TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN LAND PROJECT

SDG : 16E157

METHOD SW7471B
MERCURY BY COLD VAPOR

A total of five (5) soil samples were received on 05/18/16 to be analyzed for Mercury by Cold Vapor, 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 (ICV). Continuing calibration (CCV) verifications were carried on a frequency specified by the project. All calibration requirements were within acceptance criteria.

Method Blank
Method blank was prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed. Mercury was not detected in HGE015SB. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. HGE015SL/HGE015SC was within LCS limits. Refer to LCS summary form for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed. Mercury was within MS QC limits in E157-01M/E157-01S. Refer to Matrix QC summary form for details. 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. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met.

Appendix 1: SUMMARY OF QUALITY CONTROL PROCEDURE

PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Initial multipoint calibration	Daily initial calibration prior to sample analysis	Correlation coefficient r ≥ 0.995 for linear regression	Correct the problem then repeat initial calibration		
Initial calibration verification (second source)	Daily after initial calibration	Analyte within ± 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 ± 10% 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 > 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	%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≤ 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 (~5x the LOQ after dilution), otherwise perform MSA.		
Dilution Test	When Post-Digestion Spike fails and analyte concentration is sufficiently high (~5x the LOQ after dilution)	Within ± 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
EPA 7471B

SOP: EMAX-7471 Rev. 8
Conc Unit: mg/Kg
Sample Amount(gm): 0.6

Extraction date: 1/8/2016
Analysis date: 1/8/2016
Extracted and Analyzed by: N. Tan

PARAMETER	HGA004SL	HGA004SC	HGA005SL	HGA005SC	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	M47A003012	M47A003013	M47A003039	M47A003040							
Mercury	0.407	0.398	0.398	0.408	0.417	0.403	97	0.005	1.3	80 - 120	PASSED

7471FS: SAMPLE PREPARATION LOG

EMAX LABORATORIES, INC.

DIGESTION LOG *for* MERCURY

Page 1

Note: For samples, relevant QCs/Standards digested, refer to attached digestion sequence.

Comments:

Digestion Vessel Lot #:

Book #: E47-102

Batch No.:

Matrix:

SOP #	Rev. #
☐ EMAX-7470	8
☐ EMAX-7471	9
☐ EMAX-245.1	4
☐ EMAX-	

Standards	ID	Conc. (µg/L)	Amount Added (ml)
ICAL			
CCV			
ICV			
LCS/MS			

Reagent	ID / Lot #
HNO_3	
HCl	
H_2SO_4	
$KMnO_4$	
$K_2S_2O_8$	
$NH_2OH{\bullet}HCl{\bullet}NaCl$	
$SnCl_2$	
Silica Sand	
Reagent Water	
pH strip 0-14	

Digestor ID/ Temp (°C)

Thermometer ID/LOC:

Thermometer ID/LOC:

Pipette ID:

☐ H_2SO_4 dispenser checked @ 2.5 ml with Class A graduated cylinder

☐ HCl dispenser checked @ ___ ml with Class A graduated cylinder

☐ HNO_3 dispenser checked @ ___ ml with Class A graduated cylinder

Prepared By:

Standard Added By: Witnessed By:

7471FA: ANALYTICAL RUN LOG

EMAX LABORATORIES, INC.

ANALYSIS RUN LOG
for
MERCURY

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Start Date: Start Time:

End Date: End Time:

Comments:

Book #: 447-085

Instrument No.: 47

Analytical Sequence/Batch:

Method File:

Micropipette ID: ☐ ICP DOE-1

Micropipette ID: ☐ HG-03

Micropipette ID: ☐ HG-04

Micropipette ID: ☐

SOP #	Rev. #
☐ EMAX-7470	
☐ EMAX-7471	
☐ EMAX-245.1	
☐ EMAX-	

STANDARDS ID
S1
S2
S3
S4
S5
S6
CCV
ICV
LCS
Analytical Spike

Analyzed By:

Date:

7471FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

instrumetnt No: 47 Month Year: 201_ Book # M47-010

DAQ TEST							
Sample: 700,000 - 1,720,000							
Hg Lamp Volt: 8.0-9.5							
GAS CONTROL TEST							
Argon Input Pressure: 75-90 psi							
Power Supply Voltage: 5V+/-0.25, +12V+/-0.5, -12V+/-0.5							
Gas: 0 - 1.0 LPM							
Pump Rate: 2 - 15 ml/min							
Instrument Warm-up Minimum 5 min							
Peristaltic Pump							
Tubings Pump/Capillary							
Fume Hood							
Recirculant Bottle							
Rinse Solution							
Computer Date/Time							
Printer Paper							
Analyst Initial							
Trouble Shooting							
Problem(s) Encountered							
Return To Specs (Analyst Initial)							

Code		Code	Corrective Action

SOP QA REVIEW FORM

EMAX-8260	Rev. 10	VOLATILE ORGANICS BY GC/MS
SOP No.	Revision Number	Title

SECTION	✓	COMMENTS
Scope & Application	✓	
Summary of Method	✓	
Detection Limits	✓	
Dynamic Range	✓	
Sample Holding Time & Preservation	✓	
Associated SOPs	✓	
Safety	✓	
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 necessary.

Reviewed By [signature] Date: 6/27/16

FAKIATA MADAMBA

STANDARD OPERATING PROCEDURES

VOLATILE ORGANICS BY GC/MS

SOP No.:	EMAX-8260	Revision No.	10	Effective Date:	06-Jun-14
Prepared By:	Souzan Greas			Date:	06-04-14
Approved By:	Kenette Pimentel QA Manager			Date:	06.04.14
Approved By:	Caspar Pang Laboratory Director			Date:	06-04-14

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)**

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.[1]

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 $Na_2S_2O_3$ to pH 4-5. Samples received unpreserved must be analyzed with 3 days from sampling date[2].

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.

[1] Referenced from SW846 Method 5030B, Section 6.1.
[2] Reference: 40CFR Table 11 Footnote 10

- 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 ≤ 6°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

Column	RTX 502.2 (0.32 mm x 60 m), 1.8 µm thickness or equivalent after verification that the four gases (chloromethane, bromomethene, 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)**

9.4.1. 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.*

9.5.1.2. 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.*

9.7.1.2. 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 ≥ 2 and residual chlorine ≥ 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.

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 ± 0.06 RRT units.

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 ≤ 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 ≤ 15% and correlation coefficient of ≥ 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 ≤ 15% and correlation coefficient of ≥ 0.995.

10.3.3.6.5. It may be appropriate to force the regression through zero for specific analytes[3]. 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 ± 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%).

[3] SW846 Method 8000B, Section 7.5.3

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 ± 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 ± 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 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.[4]

10.4.3.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification[5] (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%.

[4] SW846 Method 8260B, Section 7.6.1.4

[5] Library search is performed only when indicated in the PSR.

- 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)**

$$RRF = \frac{A_X C_{IS}}{A_{IS} C_X}$$ 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_m)**

$$RRF_m = \frac{\sum RRF}{n}$$ 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**

$$y = ax + b$$ 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**

$$y = ax + b$$ 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:

$$x_a = \sum \left[x(1/x) / \sum (1/x) \right]$$
$$y_a = \sum \left[y(1/x) / \sum (1/x) \right]$$

or $x_a = \sum\left[x(1/x^2)/\sum(1/x^2)\right]$

$y_a = \sum\left[y(1/x^2)/\sum(1/x^2)\right]$

b – x0 = intercept of the line

$b = y_a - a * x_a$

10.5.1.5. **Calculate Inverse Quadratic**

$y = ax^2 + bx + c$

where:

y – Resp_Ratio =**x0** + **x1** * Amt_Ratio + **x2** * (Amt_Ratio)^2

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_i = amount ratio= Conc of Std/Conc of IS

Y_i = response ratio=Resp of Std/Resp of IS

Wi= 1/X)/SUM(1/X)

<X>= SUM(W_i*X_i)

<Y>= SUM(W_i*Y_i)

<XX>= SUM(W_i*(X_i)^2)

<XXX>= SUM(W_i*(X_i)^3)

<XXXX>= SUM(W_i*(X_i)^4)

<YY>= SUM(W_i*(Y_i)^2)

<XY>= SUM(W_i*X_i*Y_i)

<XXY>= SUM(W_i*(X_i)^2*Y_i)

<Yd2>= SUM((Y_i-<Y>)^2*W_i)

Ye= x0+x1*X_i+x2*X_i^2-<Y>)

<Ye2>= SUM(Ye^2*W_i)

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>

r2= <Ye2>/<Yd2>

ccf2= $(r2)^{1/2}$

10.5.1.6. **Calculate the Standard Deviation (SD)**

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}} \qquad \text{Eq.-10.5.1.6}$$

where:

x_i – Result at i^{th} measurement

$\bar{x}$ – Mean of the n measurements

n – Number of measurements

10.5.1.7. **Calculate the % relative standard deviation (%RSD)**

$$\%RSD = \frac{SD}{RRF_m} * 100\% \qquad \text{Eq.-10.5.1.7}$$

where:

SD – Standard deviation

RRF_m – Average response factor

10.5.1.8. **Calculate the relative retention time (RRT)**

$$\text{RRT} = \frac{\text{Retention Time of the Analyte}}{\text{Retention Time of the Internal Standard}} \qquad \text{Eq.-10.5.1.8}$$

10.5.2. Calibration Check/Continuing Calibration

10.5.2.1. **Calculate Percent Difference (%D)**

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \qquad \text{Eq.-10.5.2.1}$$

where:

RRF_c – Response factor from continuing calibration standard
RRF_m – Average response factor

10.5.2.2. **% Drift**

$$\%Drift = \frac{[found\ Conc. - true\ Conc]}{true\ Conc.} * 100\% \qquad \text{Eq.-10.5.2.2}$$

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**

$$Concentration\ (ug/L) = \frac{(Ax)(Is)}{(Ais)(RRF_m)}\ x\ DF \qquad \text{Eq.-10.5.3.2}$$

where:

A_x – Area of characteristic ion for the compound to be measured
I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor = $\frac{purge\ volume\ in\ ml\ \ (5\ ml\ or\ 25\ ml)}{sample\ \ amount\ \ in\ ml}$

10.5.3.3. **Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(Ax)(Is)}{(Ais)(RRF_m)(DW)}\ x\ DF \qquad \text{Eq.-10.5.3.3}$$

where:

A_x – Area of characteristic ion for the compound to be measured
I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor = $\frac{5\ g}{(sample\ \ amount\ \ in\ g)}$
DW – % solid = $\frac{100 - \%moisture}{100}$

10.5.3.4. **Extracted Soil/Sediment Samples (Dry weight basis)**

$$Concentration\ (ug/kg) = \frac{(Ax)(Is)}{(Ais)(RRF_m)(DW)}\ x\ DF \qquad \text{Eq.-10.5.3.4}$$

where:

A_x – Area of characteristic ion for the compound to be measured

I_s – Concentration of internal standard added in µg/L
A_{is} – Area of characteristic ion for the internal standard
RRF_m – Average response factor
DF – Dilution factor

$$\frac{(purged\ \ volume\ \ in\ \mu L)(5\,g)}{(extract\ \ aliquot\ \ in\ \mu L)(sample\ \ amount\ \ in\ g)}$$

DW – % solid = $\frac{100 - \%moisture}{100}$

10.5.4. Alternatively, the regression line (area ratio of Ax/Ais 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 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. Method Proficiency

10.5.6.1. **Percent Recovery**

$$\%\,\mathrm{Recovery} = \frac{(C_f - C)}{C_s} \times 100 \qquad \text{Eq.-10.5.6.1}$$

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)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \qquad \text{Eq.-10.5.6.2}$$

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.

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-DM01and 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.

- ✓ Check retention time of each IS to the ICAL midpoint. They should be within ± 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						✓

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 ± 0.06 RRT units.

11.1.7. Verify internal standard (IS) for quantitative accuracy and that its Retention time is within ± 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:

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:

- 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 ≥ 2 and/or residual chlorine ≥ 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.

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.

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 (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.

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. 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**

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

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
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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	LCS/MS	WATER (µg/L)			SOIL (µg/Kg)		
	PRIMARY	SECONDARY			1	2	3	4	5	6	7	8	EV(µg/L)	EV(µg/L)	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

UNCONTROLLED WHEN PRINTED

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

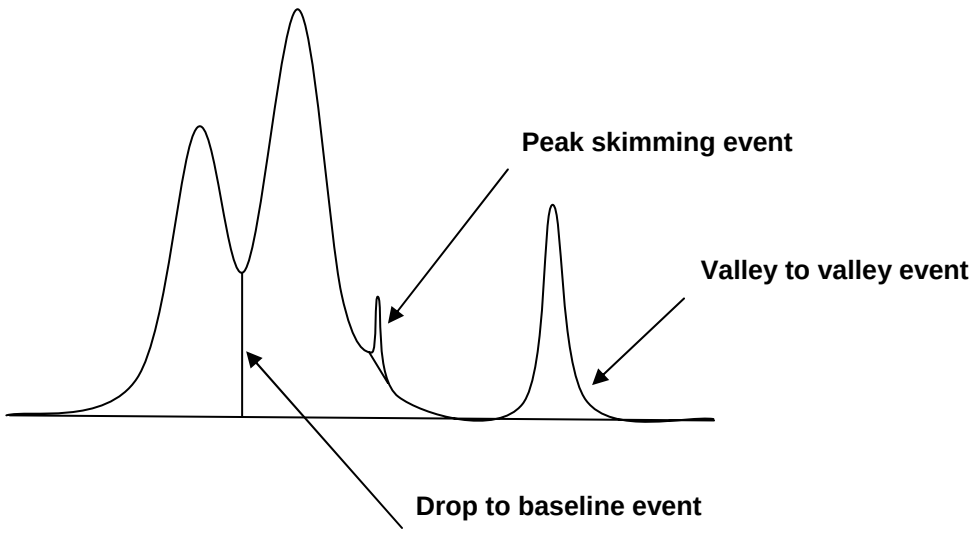
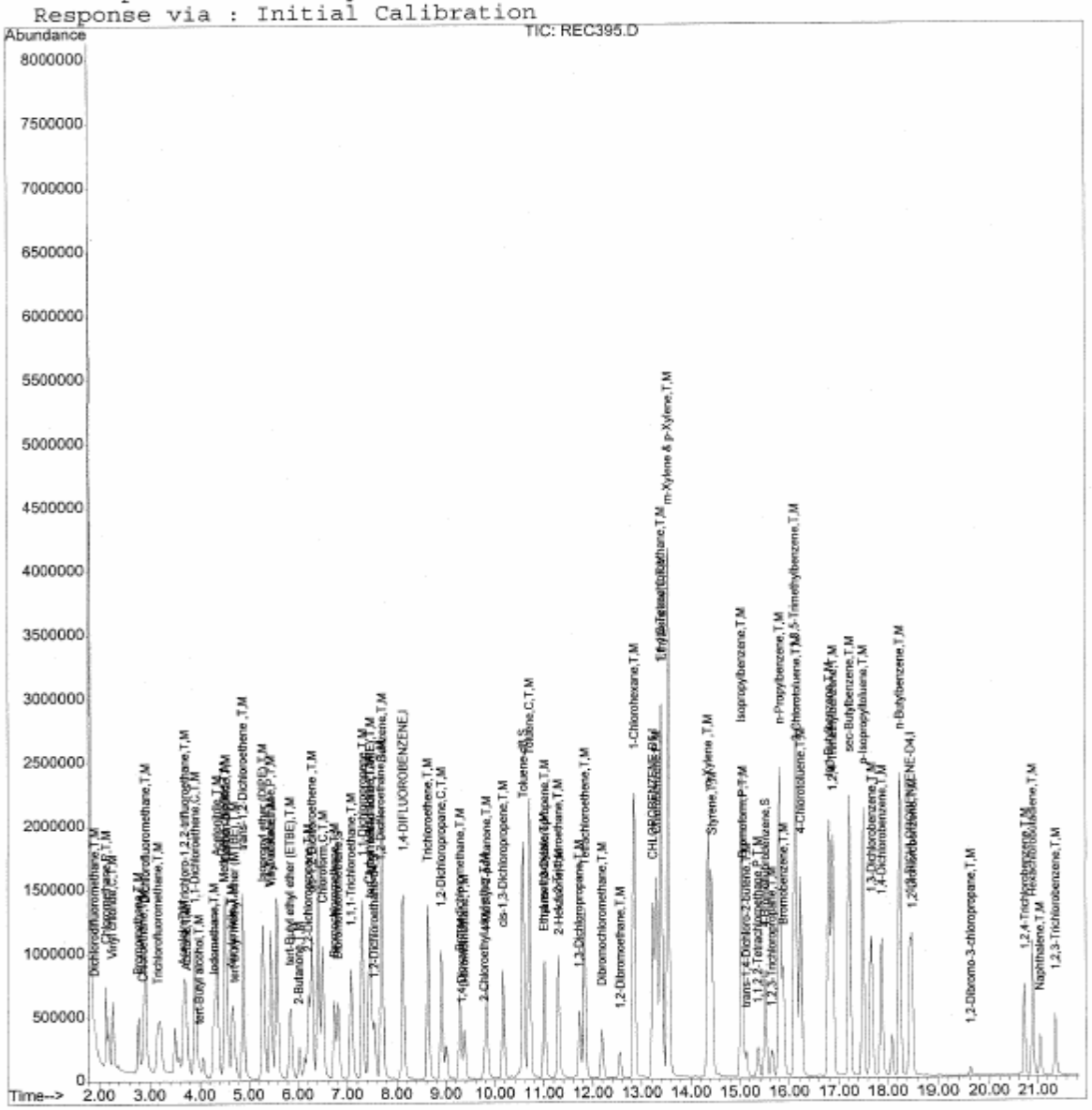


Figure 2: **TYPICAL CHROMATOGRAM**

Quantitation Report

Data File : D:\HPCHEM\1\DATA\11E25\REC395.D Vial: 2
Acq On : 25 May 2011 1:40 pm Operator: AS
Sample : IV067E2402 10ppb Inst : TO67
Misc : 10ppb 8260/50ppb KET-ACR-ACN-TBA Multiplr: 1.00
MS Integration Params: LSCINT1.P
Quant Time: May 25 16:32 2011 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



REC395.D VO67E24.M Wed May 25 16:38:57 2011 Page 4

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 07:57 RCB018	.5 08:31 RCB019	1 09:05 RCB020	2 09:39 RCB021	5 10:11 RCB022	10 10:45 RCB023	20 11:18 RCB024	30 11:52 RCB025	50 12:25 RCB026	100 12:58 RCB027	Av_RRF	%_RSD	Av_Rt_M
	1	1,4-DIFLUOROBENZENE	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
5 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
5 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
2 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	4.10[illegible]	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 = xo + x1 * Amt_Ratio

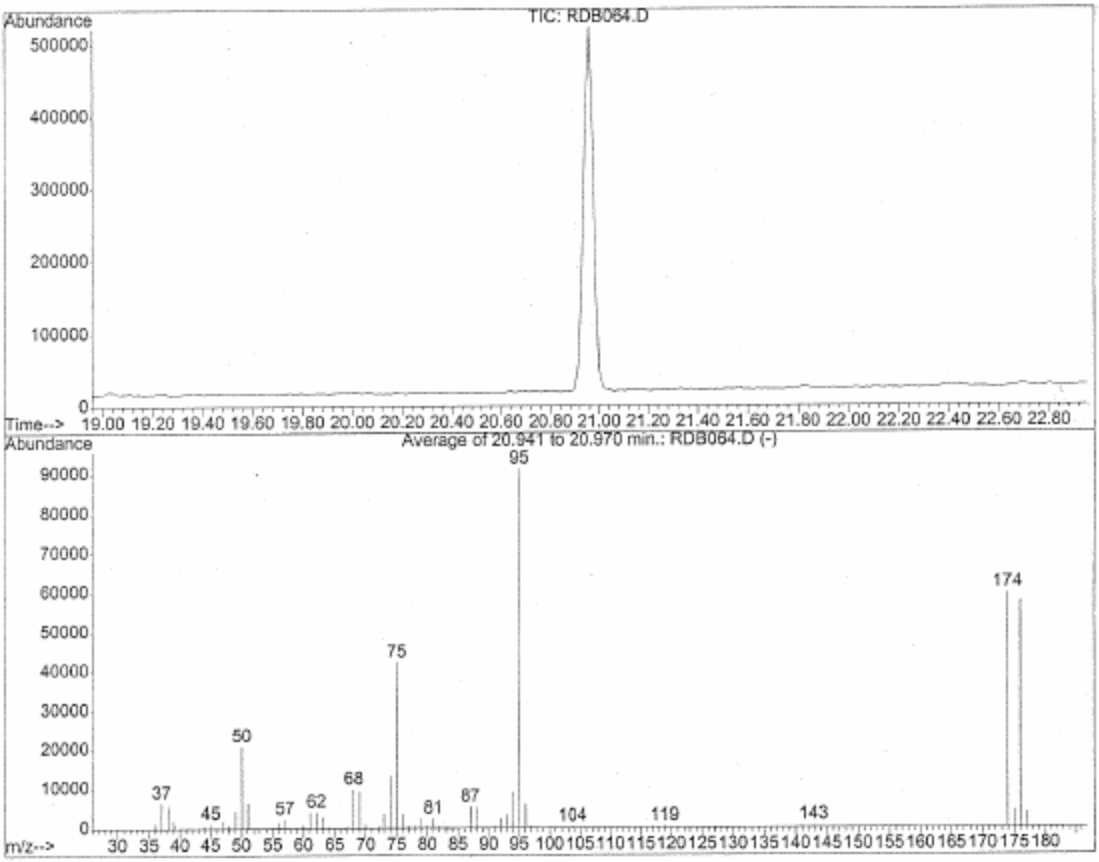
IDX	Parameter	x0	x1	CCF
14	Acetone	0.00872	0.02772	0.9996

Figure 4: **TYPICAL INSTRUMENT PERFORMANCE CHECK (TUNING)**

BFB

Data File : D:\HPCHEM\1\DATA\11D07\RDB064.D
Acq On : 7 Apr 2011 7:43 pm
Sample : BFB03D04
Misc : T/CHECK
MS Integration Params: 524INT.P
Method : D:\HPCHEM\1\METHODS\VO03D07.M (RTE Integrator)
Title : METHOD 8260 25mL

Vial: 2
Operator: MW
Inst : TO03
Multiplr: 1.00



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

RDB064.D VO03D07.M Fri Apr 08 09:01:53 2011

Figure 5: TYPICAL INSTRUMENT PERFORMANCE CHECK (TUNING) SUMMARY

5A
VOLATILE ORGANIC INSTRUMENT PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

Lab Name: EMAX Inc — Contract: CLEAN WATER PROJECT
Lab Code: EMXT — Case No.: — SAS No.: — SDG No.: YYMNNN
Lab File ID: RDC521 — BFB Injection Date : 04/29/14
Instrument ID: 67 — BFB Injection Time : 09:52
GC Column:RTX502.2ID:0.25mm (mm) — Heated Purge: (Y/N) N

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	20.69
75	30.0 - 60.0% of mass 95	45.82
95	Base peak, 100% relative abundance	100.00
96	5.0 - 9.0% of mass 95	6.39
173	Less than 2.0% of mass 174	0.00(0.0)1
174	Greater than 50% of mass 95	70.43
175	5.0 - 9.0% of mass 174	5.04(7.2)1
176	95.0 - 101.0% of mass 174	67.25(95.5)1
177	5.0 - 9.0% of mass 176	4.57(6.8)2

1-Value is % mass 174 — 2-Value is % mass 176

THIS CHECK APPLIES TO THE FOLLOWING SAMPLES, MS, MSD,BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
1	VSTD010	CVO67D0914	RDC523	04/29/14	11:14
2	MBLK1W	VO67D21B	RDC527	04/29/14	13:15
3	LCS1W	VO67D21L	RDC524	04/29/14	11:44
4	LCD1W	VO67D21C	RDC525	04/29/14	12:15
5	XXX-59XX001	MNNN-02	RDC534	04/29/14	16:55
6	XXX-59XX001MS	MNNN-02M	RDC535	04/29/14	17:26
7	XXX-59XX001MSD	MNNN-02S	RDC536	04/29/14	18:57

Figure 6: TYPICAL INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

8A
VOLATILE INTERNAL STANDARD AREA AND RT SUMMARY

Lab Name : EMAX INC.
Lab Code : EMXT
Lab File ID : RDC168
Instrument ID: 67
GC Column : RTX502.2ID:0.25mm (mm)

Project : CLEAN WATER PROJECT
SDG No. : YYMNNN
Date Analyzed : 04/09/14
Time Analyzed : 18:54
Heated Purge : No

		IS1(DBF)		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

Client	: XYZ INC.	Date Collected:	04/28/14
Project	: CLEAN WATER PROJECT	Date Received:	04/29/14
Batch No.	: YYMNNN	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
CHLOROBENZENE	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-DICHLOROBENZENE	ND	1.0	0.10	0.20
1,3-DICHLOROBENZENE	ND	1.0	0.11	0.20
1,4-DICHLOROBENZENE	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.: YYMNNN
METHOD: SW5030C/8260B

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.: YYMNNN
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

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	2nd 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 ≥ 0.1 for Bromoform, Chloromethane and 1,1-Dichloroethane RF ≥ 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane CCCs: RSD ≤ 30% for the following analytes: Chloroform, 1,1-DCE, 1,2-DCP, Ethylbenzene, Toluene and Vinyl Chloride. 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 r ≥ 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 ± 20% of expected value except for the following compounds due to erratic chromatographic behavior: Bromomethane, Chloroethane, Chloromethane, Dichlorodifluoromethane but must be within ± 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 ± 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 ≤ 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 ± 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 > 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	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		
Comments: 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

Unit: µg/L
Sample Amount(ml): 25
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12
Analyzed by: D. Nguyen

PARAMETER	RAY133	RAY134	RBY004	RBY005	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

Appendix 2 (cont.): DEMONSTRATION OF CAPABILITY for 25 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 25
Sample Purge(ml): 25

Date Analyzed: 01/31 & 02/01/12
Analyzed by: D. Nguyen

PARAMETER	RAY133	RAY134	RBY004	RBY005	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
1,1,2,2-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-Trichloro1,2,2-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

Unit: µg/L
Sample Amount(ml): 5
Sample Purge(ml): 5

Date Analyzed: 01/07/13 & 01/08/13
Analyzed by: C. Mendoza

PARAMETER	RAN044 VSF4A07L	RAN045 VSF4A07C	RAN058 VSF4A08L	RAN059 VSF4A08C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
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	13.989	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

Appendix 3 (cont.): DEMONSTRATION OF CAPABILITY for 5 ml

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/L
Sample Amount(ml): 5
Sample Purge(ml): 5

Date Analyzed: 01/07/13 & 01/08/13
Analyzed by: C. Mendoza

PARAMETER	RAN044 VSF4A07L	RAN045 VSF4A07C	RAN058 VSF4A08L	RAN059 VSF4A08C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
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-Trichloro1,2,2-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

Appendix 4: DEMONSTRATION OF CAPABILITY for 5 g

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/Kg
Sample Amount(g): 5
Sample Purge(ml): 5

Date Analyzed: 03/20 & 03/21/12
Analyzed by: C. Mendoza

PARAMETER	RCP061 VO02C04L	RCP062 VO02C04C	RCP086 VO02C06L	RCP087 VO02C06C	True Value	Ave. Conc.	Ave. % Rec.	Std. Dev.	RSD	QC Criteria	Comments
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

Appendix 4 (cont.): DEMONSTRATION OF CAPABILITY for 5 g

DEMONSTRATION OF CAPABILITY
METHOD: SW 8260

Unit: µg/Kg
Sample Amount(g): 5
Sample Purge(ml): 5

Date Analyzed: 03/20 & 03/21/12
Analyzed by: C. Mendoza

PARAMETER	RCP061	RCP062	RCP086	RCP087	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-Trichloro1,2,2-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

8260FS: **SAMPLE PREPARATION LOG**

EMAX LABORATORIES, INC.

PRESERVATION LOG FOR VOLATILES

VP-xls

PrepBatchID	Lab Sample ID	W1 (g)	DateTime	Wf (g)	DateTime	ExpWs(g)	Ws(g)	PrepEFactor	Comments

☐ EMAX-5035 Rev. ☐ Balance Calibration Check performed prior to use Surrogate ID: Prepared By:

☐ Encore+$NaHSO_4 \cdot H_2O$ ☐ BalanceID 25451062 BlankSoil Lot#: Standard Added By:

☐ Frozen Sample ☐ BalanceID 25650797 NaHSO4H2O: Witnessed By:

☐ Pre-weighed vial Expected Ws(g): Extract Rcvd By:

☐ Volumetric dispenser was checked with 5-ml volumetric flask Sample Location:

W1=Weight of Vial + Stirrer+NaHSO4•H2O+Solvent Wf=W1 + Sample

8260FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC

Page 1

ANALYSIS LOG FOR VOLATILES

SOP ☐ EMAX-8260 Rev.No. _ ☐ EMAX-624 Rev.No. _ ☐ EMAX-8260SIM Rev.No. _ ☐ EMAX-TCPSIM Rev.No. _ ☐ EMAX-M8260SIM Rev.No. _ ☐ EMAX-8260C Rev. No. _

Start Date: ☐ 5-mL Purge ☐ 10-mL Purge ☐ 25-mL Purge

Book # A06 -053

BATCH _____

Sample Prep ID	Data File Name	Lab Sample ID	Sample Amount	DF	Matrix W pH ≤2	Matrix W O_2 <3ppm	Matrix S	Notes
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

Instrument No.	06
INITIAL CALIBRATION REFERENCE	
DATE	
ICAL ID	

STANDARDS			
NAME	ID	Amount (µL)	Conc. (mg/L)
DCC			
DCC			
DCC			
DCC			
EFB			
IS/SURR			
ICV/LCS			
ICV/LCS			
ICV/LCS			
ICV/LCS			
Data File Folder			
	LOT #		
pH strip			
Chlorine strip			
Methanol			
$NaHSO_4$			
Reagent Water			
Sand			

Electronic Data Archival Location	Date
HPCHEM_VOA/TO06	

Comments: _____

☐ Refer to sample weight log

Analyzed By: _____

Date Disposed: _____ Disposed By: _____

8260FM: INSTRUMENT MAINTENANCE LOG

EMAX LABORATORIES, INC

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 05 Month: Year: 201_ Book # : MD3-005

DATE						
Bake Trap (210°C-260°C)						
Column Head Pressure (2-60 psi)						
Injector Port Temperature (160°C-220°C)						
Interface Temperature (250°C-280°C)						
Verify Computer Date and Time						
Check Gas Pressure (Daily)						
Check IS/Surrogate auto injection vial as appropriate (Daily)						
Check the foreline pump oil level (Weekly)						
AutoTune the MSD (As needed)						
Analyst's initial						
Trouble Shooting						
Problem(s) Encountered						
Return-to-Control (Analyst Initial)						

Code	Problems Encountered	Code	Corrrective Action

STANDARD OPERATING PROCEDURE

DISSOLVED GASES

SOP No.:	EMAX-RSK175	Revision No.	3	Effective Date:	04-Apr-16
Prepared By:	Tu Nisamaneepong			Date:	04-04-16
Approved By:	Kenette Pimentel QA Manager			Date:	04-04-16
Approved By:	Caspar Pang Laboratory Director			Date:	04-04-16

Control Number: RSK175-03-

1.0 SCOPE AND APPLICATION

1.1. This method is used to determine dissolved methane, ethane, ethane, propane and acetylene in aqueous samples. This SOP is an adaptation of Dissolved Oxygen and Methane in Water by GC Headspace Equilibrium Technique published by R.S. Kerr Research Laboratory, commonly known as RSK 175.

1.2. This method is also used for carbon dioxide determination using an unpreserved sample and a Thermal Conductivity Detector (TCD).

1.3. This method is also amenable to propene, isobutene and any other gases of similar structure, upon meeting all quality control criteria.

2.0 SUMMARY OF METHOD

2.1. A known volume of inert gas is injected into the sample contained in a septum cap vial, which subsequently displaces the same volume of sample into an injected empty disposable syringe. The injected gas is allowed to equilibrate with the sample by wrist mixing. Subsequently, a known amount of gas is aspirated from the headspace by a tight syringe with a locking device is analyzed for the target gases by GC with FID detector.

2.2. **Interferences**

2.2.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to its 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.

2.2.3. 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 of DL, LOD and LOQ.

3.1.2. Below are the established limits.

Parameter	DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Methane	0.17	0.34	0.68
Acetylene	0.275	0.55	1.10

Parameter	DL (µg/L)	LOD (µg/L)	LOQ (µg/L)
Ethylene	0.3	0.6	1.19
Ethane	0.32	0.64	1.27
Propane	0.465	0.93	1.86
Carbon Dioxide	300	600	1200

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). All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.

4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (See Section 9.3.1). All samples analyzed below this concentration are considered "under-range" and shall require a lower dilution factor for proper quantitation.

5.0 SAMPLE HOLDING TIME AND PRESERVATION

5.1. **Preservation**

5.1.1. Water samples are received in 40 mLl vials with teflon-lined septa with zero headspace. The size of bubble caused by degassing upon cooling the sample should not exceed 6 mm.

5.1.2. Except for samples requiring carbon dioxide analysis, samples are preserved with concentrated HCl to pH < 2. Samples for carbon dioxide analysis must be analyzed from a sample with no chemical preservative.

5.1.3. All samples are stored at ≤ 6°C without freezing.

5.2. **Holding Time**

5.2.1. The holding time is 14 days from the time of collection.

5.2.2. For unpreserved water samples, the holding time is 7 days from collection date.

6.0 ASSOCIATED SOPs

6.1. EMAX-QA04 Detection Limit (DL)

6.2. EMAX-QA08 Corrective Action

6.3. EMAX-QC02 Analytical Standard Preparation

6.4. EMAX-QC05 Calibration of Thermometers

6.5. EMAX-QC06 Volumetric Labware and Micropipette Verification

6.6. EMAX-DM01 Data Flow and Review

6.7. EMAX-SM02 Sample Receiving

6.8. EMAX-SM03 Waste Disposal

6.9. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

7.1. Read all SDS for 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 copious amount of water. If irritations or any discomfort related to the incident 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	Varian 3400 with FID, or equivalent For CO_2, HP 5890 Series II w/ TCD or equivalent
Column	Carboxen 30 m x 0.53 mm ID, 5 µm thickness (Supelco or equivalent)
Gas	Ultra-high purity helium; ultra-high purity hydrogen
Syringes	Standard Syringe – 25 µL, 100 µL, 250µL, 5 mL Luerlock gas tight syringe with shut-off valve, or equivalent Helium Syringe – 3 mL and 5 mL Luerlock gas tight syringe with shut-off valve, or equivalent Displacement Syringe – 5 mL Luerlock disposable syringe
Microsyringes	25, 100, and 250 µL with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes
Data System	EZ-Chrom, HP Chem Station, or equivalent
Thermometer	0-100°C

8.2. **Chemicals and Reagents**

8.2.1. Solvent: Organic-free water

9.0 STANDARDS

9.1. **Stock Standard**

9.1.1. Purchase Stock Standards as certified solutions as listed below (or equivalent).

Standard Name	Source	Concentration (mg/L, v/v)	Intended Use
Gas mixture of ethane, ethylene and methane, propane, acetylene and CO_2 in Helium	Scotty Gases	500, 10%	ICAL, DCC*

Gas mixture of ethane, ethylene and methane, propane, acetylene and CO_2 in Helium	Matheson	500, 10%	ICV, LCS, MS*

*Note: * Stock standards may be interchanged to meet the intended use during calibration.*

9.2. **Initial Calibration Standard (ICAL)**

9.2.1. Prepare the ICAL points at concentration levels as suggested in the table below to a final volume of 4 mL Helium.

Standard		**1**	**2**	**3**	**4**	**5**	**6**
Preparation of ICAL Standards							
Primary Stock Std. (500 ppm) Aliquot (mL)		0.08	0.4	1.0	1.6	3.0	4.0
Solvent		Helium	Helium	Helium	Helium	Helium	-
Final Volume (mL)		4	4	4	4	4	4
Final Concentrations							
C_2H_2	(µL/L)	10	50	125	200	375	500
	(µg/L)*	1.1	5.5	14	22	41	55
CH_4	(µL/L)	10	50	125	200	375	500
	(µg/L)*	0.68	3.4	8.5	14	26	34
C_2H_4	(µL/L)	10	50	125	200	375	500
	(µg/L)*	1.2	6	15	24	45	60
C_2H_6	(µL/L)	10	50	125	200	375	500
	(µg/ml)*	1.3	6.4	16	26	48	64
C_3H_8	(µL/L)	10	50	125	200	375	500
	(µg/L)*	1.9	9.4	23	37	70	94

Note: Conversion of concentration from µL/L to µg/L is shown in Eq. 10.5.1.1 with V_h/V_w = 4/39 and T = 21 ^oC.

9.2.2. For carbon dioxide calibration, prepare the ICAL points at concentration levels as suggested in the table below to a final volume of 4 ml Helium.

Standard		1	2	3	4	5	6
Preparation of ICAL Standards							
Primary Stock Std (10%) Aliquot (mL)		0.25	0.5	1.0	2.0	3.0	4.0
Solvent		Helium	Helium	Helium	Helium	Helium	-
Final Volume (mL)		4	4	4	4	4	4
Final Concentration							
CO_2	(µL/L)	6250	12500	25000	50000	75000	100000
	(µg/L)*	1200	2300	4700	9300	14000	19000

Note: Conversion of concentration from µL/L to µg/L is shown in Eq. 10.5.1.1 with V_h/V_w = 4/39 and T = 21 °C.

9.3. **Initial Calibration Verification Standard (ICV) & LCS/MS Spike Standard**

9.3.1. Prepare ICV standard using secondary source stock standard to final volume of 4 mL as suggested below.

Preparation			Final Concentration				
Stock Std Aliquot (ml)	**Solvent**	**Final Vol. (ml)**	**C_2H_2**	**CH_4**	**C_2H_4**	**C_2H_6**	**C_3H_8**
1.6	Helium	4	200 µL/L	200 µL/L	200 µL/L	200 µL/L	200 µL/L
			22 µg/L	13.6 µg/L	23.7 µg/L	25.4 µg/L	37.3 µg/L

9.3.2. For carbon dioxide, prepare as suggested below.

Preparation			Final Concentration
Stock Std Aliquot (ml)	**Solvent**	**Final Vol. (ml)**	**CO_2**
2	Helium	4	50000 µL/L
			9300 µg/L

9.4. **Continuing Calibration Standard (DCC)**

9.4.1. Prepare ICV standard using primary source stock standard to final volume of 4 mL as suggested below.

Preparation			Final Concentration				
Stock Std Aliquot (ml)	**Solvent**	**Final Vol. (ml)**	**C_2H_2**	**CH_4**	**C_2H_4**	**C_2H_6**	**C_3H_8**
1.6	Helium	4	200 µL/L	200 µL/L	200 µL/L	200 µL/L	200 µL/L
			22 µg/L	13.6 µg/L	23.7 µg/L	25.4 µg/L	37.3 µg/L

9.4.2. For carbon dioxide, prepare as suggested below.

Preparation			Final Concentration
Stock Std Aliquot (ml)	**Solvent**	**Final Vol. (ml)**	**CO_2**
2	Helium	4	50000 µL/L
			9300 µg/L

10.0 PROCEDURES

10.1. **Sample Preparation**

10.1.1. Let the samples sit on a water bath for at least an hour or until the water bath equilibrates with the room temperature (21 ± 2 °C). For LCS, prepare reagent water in 4 mL vial. If MS is required, prepare an extra vial of designated sample.

- Check that the thermometer has a valid calibration. Record the thermometer ID and temperature of the water bath in the sample preparation log.

10.1.2. Place the sample vial upside down and secure with a three finger clamp. Insert the sample displacement syringe, set for dead volume, into the sample by penetrating the septum to expose about one centimeter of the syringe needle into the sample.

10.1.3. From a freshly helium-filled tedlar bag, using the helium syringe, measure 4mL of helium into a gas tight Luerlock syringe, lock then insert into the vial septum. For LCS and MS, spike LCS/MS standard as suggested in Sec. 9.3.

10.1.4. Open the syringe lock and inject the helium into the sample. As the helium is injected, sample is displaced from the vial into the empty syringe, creating a headspace equivalent to the volume of helium injected.

10.1.5. Check the pH of the discarded sample and record in the sample preparation log.

10.1.6. Shake the vial for five minutes by quick wrist motion to allow gas to equilibrate between the liquid and gas phases.

10.1.7. The gas contained in the vial headspace is ready for analysis. Perform the analysis immediately upon completion of a preparative batch.

10.2. **Instrument Parameters**

10.2.1. Gas Chromatography Condition (FID)

Gases Setting:	
H_2	30 psi
Helium	30 psi
Air	120 psi
GC Setting	
Signal	1.43-1.56 mV
Injection Temperature	200 °C
Detector Temperature	225 °C
Injection Volume	100 µL
Head Pressure	12 psi
Carrier & Make-up gases + Air	300 mL/min

Temperature Program	Temperature (°C)	Rate/min (°C)	Time (min)
Column	80	0	0
	220	30	5

10.2.2. Gas Chromatography Condition CO_2 (TCD)

Gases Setting:

H_2	30 psi
Helium	30 psi
Air	120psi
GC Setting	
Injection Temperature	230°C
Detector Temperature	230°C
Injection Volume	100 µL
Head Pressure	10 psi

Temperature Program	Temperature (°C)	Rate/min (°C)	Time (min)
Column	80	0	1
	225	24	1

10.3. **Calibration**

10.3.1. Initial Calibration

10.3.1.1. Carefully fill a 40 mL VOA vial with reagent water. Care should be taken to avoid any trapped bubbles.

10.3.1.2. Setup the vial as described in Sec. 10.1.1.

10.3.1.3. Using a Luerlock gas tight syringe measure the desired amount of standard (refer to Sec. 9.2) and lock it. Using the helium syringe, measure the corresponding amount of helium to make a total of 4mL final volume of gas (standard plus helium) and lock it.

10.3.1.4. Insert the standard syringe and the helium syringe into the vial septum. Simultaneously inject the standard and helium (as described in Sec. 9.2.1) into the vial to displace 4mL of reagent water into the displacement syringe. Lock the syringe after injection was completed to prevent sample backflow.

10.3.1.5. Remove the syringes and shake the vial for five minutes by quick wrist motion to allow gas to equilibrate between the liquid and gas phases.

10.3.1.6. With the vial positioned upright, withdraw 100 µL gas from the headspace and analyze by GC.

10.3.1.7. Calculate the mean calibration factor and the relative standard deviation (%RSD) according to Eq. 10.5.1.2 and Eq. 10.5.1.5.

10.3.1.8. Refer to Appendix 1 for acceptance criteria. If results are non-compliant, refer to Sec. 12 for corrective action.

10.3.2. Initial Calibration Verification

10.3.2.1. Prepare and analyze an ICV standard (refer to Sec.9.3) applying the same procedure as described in Sec.10.3.1.

10.3.2.2. Refer to Appendix 1 for acceptance criteria. If results are non-compliant, refer to Section 12 for corrective action.

10.3.3. Daily Continuing Calibration (DCC)

10.3.3.1. Analyze a continuing calibration standard (refer to Sec. 9.4) to check the validity of the ICAL, at the beginning of the batch, every 12 hours thereafter and at the end of the batch.

10.3.3.2. Calculate the percent difference using Eq. 10.5.2.

10.3.3.3. Refer to Appendix 1 for acceptance criteria. If results are non-compliant, refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. Prepare analytical sequence as suggested below.

10.4.1.1. Instrument blank – inject 100µL of helium to confirm absence of contamination from the system and the helium to be used for analysis.

10.4.1.2. DCC – to check the validity of the ICAL prior to sample analysis.

10.4.1.3. Method Blank – to confirm absence of contamination from the reagent water used for the standard preparation as well as the naturally occurring gases from the atmosphere.

10.4.1.4. LCS – Since DCC is prepared and analyzed the same way as the LCS, the objective of analyzing LCS is the same as DCC.

10.4.1.5. Samples not to exceed 12 hours

10.4.1.6. MS/MSD – as required by the project.

10.4.1.7. DCC – after 12 hours and at the end of the batch

10.4.2. Sample Result Evaluation

10.4.2.1. Check QC parameters as soon as the data is available.

10.4.2.2. When a chromatographic peak falls in the retention time window (Sec. 10.4.4) of an analyte, it is considered as qualitatively identified. Calculate the concentration according to Eq. 10.5.3.

10.4.2.3. Check the concentration of identified target analytes. If the response exceeds the calibration range, re-analyze the sample to bring the response of the target analyte within the calibration range guided by the options below.

- If less than 10% of the original sample headspace was used in the sample analyses, a smaller injection from the same headspace can be made.
- Otherwise, a new sample is prepared at an appropriate dilution.

10.4.2.4. Rule out carryover. Check sample(s) analyzed after a highly concentrated sample that they do not have carryover.

10.4.3. Manual Integration

10.4.3.1. When manual integration is necessary, follow the procedures described in EMAX-DM01.

10.4.4. Retention Time Windows

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 obtained retention time for each analyte according to Eq.10.5.1.4.

10.4.4.1.3. The width of RTW is defined by ± 3xSD.

10.4.4.2. Evaluating RTW

10.4.4.2.1. If the SD is equal to 0.0, default to the previous study until historical data is obtained to define the RTW of the current instrument.

10.4.4.2.2. For new instruments, use the established retention time of 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 the absolute retention time for each 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

10.4.4.4.1. Re-establish the RTW as described in Sec. 10.4.4.1 when 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.)

10.4.4.4.2. If the calculated new RTW is significantly narrower than the previously established RTW, default to the previously established RTW.

10.4.5. Dealing with Carryover

10.4.5.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.5.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.5.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. **Calculations**

10.5.1. Initial Calibration

10.5.1.1. Standard Concentration from µL/L to µg/L

$$C = \frac{V_H}{V_W} \times C_V \left(\frac{273}{273 + T_C} \right) \times \frac{1}{22.4} M_{wt}$$ Eq. 10.5.1.1

where:

C	–	Concentration of gas in water in µg/L
V_H	–	Head space volume (4mL)
V_W	–	Sample volume (39 mL)
C_V	–	Concentration of gas in µL/L
T_C	–	Room temperature (21°C ±2°C)
M_{wt}	–	Molecular Weight of gases:
		C_2H_2 – 26 (µg/µmol)
		CH_4 – 16 (µg/µmol)
		C_2H_4 – 28 (µg/µmol)
		C_2H_6 –30 (µg/µmol)
		C_3H_8 –44 (µg/µmol)
		CO_2 – 44 (µg/µmol)

10.5.1.2. Calculate for Calibration Factor (CF).

$$CF = \frac{R_a}{C_a}$$ Eq. 10.5.1.2

where:

R_a	–	Response for analyte measured in peak area
C_a	–	Concentration of analyte to be measured (µg/L)

10.5.1.3. Calculate for Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF}{n}$$ Eq. 10.5.1.3

where:

ACF	–	Average calibration factor
ΣCF	–	Sum of calibration factors
n	–	Number of calibration points

10.5.1.4. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$ Eq. 10.5.1.4

where:

SD	–	Standard deviation
x_i	–	Result at i^{th} measurement
$\bar{x}$	–	Mean of the n measurements
N	–	Number of measurements

10.5.1.5. Calculate for % Relative Standard Deviation (%RSD).

$$\%RSD = \frac{SD}{ACF} * 100\%$$ Eq. 10.5.1.5

where:

SD	–	Standard deviation
ACF	–	Average calibration factor

10.5.2. Calculate for Continuing Calibration Percent Difference (%D)

$$\%D = \frac{Ck - Cf}{Ck} * 100\%$$ Eq.10.5.2

where:

% D	–	Percent difference of DCC from known concentration.
Ck	–	*K*nown concentration of analyte, in µg/L
Cf	–	Concentration found, in µg/L

10.5.3. Sample Results

$$C = \frac{R_a}{ACF} * D$$ Eq.-10.5.3

where:

C	–	Concentration of analyte (µg/L)
R_a	–	Response for analyte measured in peak area
ACF	–	Average calibration factor of analyte
D	–	Dilution factor

10.5.4. Accuracy and Precision

10.5.4.1. Percent Recovery (%R)

$$\%R = \frac{C_f - C}{C_s} * 100$$ Eq. 10.5.4.1

where:

C_f – Concentration found

C – Sample concentration; for LCS, C=0

C_s – Concentration of spike

10.5.4.2. Relative Percent Difference (RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$$ Eq. 10.5.4.2

where:

C_1 – Measured concentration of the first aliquot

C_2 – Measured concentration of the second aliquot

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. Collate the reportable raw data separating the QC results from the sample results.

10.6.3. Keep all other data generated with the analytical folder marked with “For record only”.

10.6.4. Proceed to report generation applying the calculation formulas in 10.6 as appropriate.

10.7. **Report Generation**

10.7.1. Generate the method.txt file using WD1CN.exe.

10.7.2. Generate Lab Chronicle using LABCHRN1.exe

10.7.3. Generate the sample results using F1NV3C4.exe

10.7.4. Generate the QC summary using QCV3N4.exe

10.7.5. Generate case narrative using CN00.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 and re-analyses.

- Case Narrative
- Lab Chronicle
- Sample Results
- Method Blank Results
- LCS/ LCSD Summary
- MS/MSD Summary
- ICAL Summary

- ICV Summary
- DCC Summary
- Analysis Log
- Sample Preparation Log
- Non-Conformance Report (if any)

10.8. **Data Review**

10.8.1. Perform a 100% data review in accordance to EMAX-DM01and the PSR.

10.8.1.1. If any of the checkpoints below indicate a problem, re-analysis is required.

- Check calibrations for accuracy, completeness and compliance to requirements.
- Check that concentration of target analytes reported are all within calibration range.
- Check that method blank is compliant to PSR.
- Check that LCS/LCD is/are within the project QC limits.
- Check that MS/MSD are within the project QC limits.

10.8.1.2. Review that the attached logs are properly filled.

10.8.1.3. Check the generated reports against raw data. Check that the analytical data that indicares 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 analysis package for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Perform routine instrument preventive maintenance and record on the instrument-specific maintenance log (Refer to Form RSK175FM).

10.9.2. Routine maintenance ensures that all Instruments are operating under optimum condition, thus reducing the possibility of instrument malfunction that may affect data quality.

10.9.3. The table below lists suggested routine maintenance schedule.

Maintenance Activity	Description	Frequency
GC Verification	Inspect and clean syringe. Check instrument parameters to ensure normal operating conditions. Change liner as necessary. Clean injection port as necessary. Check instrument performance (e.g., Daily calibration check, instrument blank)	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

Maintenance Activity	Description	Frequency
Check Flow Path Components	Check and replace the following as necessary: tubing assembly, union, column	Once a year as necessary
Complete Inspection	Perform general inspection of the complete system. Inspect autosampler cabling and configuration setting.	Once a year
Documentation	Record all instrument maintenance performed in the instrument maintenance log.	Daily prior to analysis

11.0 QUALITY CONTROL

11.1. Method QC

11.1.1. Establish DL, LOD and LOQ prior to using this method. Refer to EMAX-QA04 for evaluation and verification.

11.1.2. All analysts performing the method should have a valid Demonstration of Capability (DOC).

11.2. Analytical Batch

11.2.1. Perform sample analysis with a valid ICAL. Analyze ICV to verify the concentration of the ICAL standards.

11.2.2. An instrument blank shall be performed prior to the first daily continuing calibration. The acceptance criteria shall be the same as the method blank.

11.2.3. Analyze DCC at a frequency and acceptance criteria as described in Appendix 1 to validate the ICAL.

11.2.4. Update the Retention Time Window at least once a year or when a major instrument repair is done.

11.3. Sample Preparation

11.3.1. All samples must be analyzed within the holding time.

11.3.2. The maximum number of original field samples in a preparation batch shall be 20 unless otherwise specified by the project.

11.3.3. Clean the purge needles thoroughly with high purity helium, after every use to ensure that there are no particulates adhering to it.

11.3.4. Prepare and analyze method blank, LCS (when required by the project), and MS/MSD (when designated by the project) for each every analytical batch applying the same analytical process as the field samples (Section 10.0).

11.3.5. Use organic-free water for method blank and LCS.

11.3.6. Analyze LCS and MS/MSD samples as specified by the project. In the absence of any project designation, a sample duplicate with detected analytes or a LCS duplicate may be analyzed to establish precision.

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 the % RSD is out of acceptance criteria, review the results and identify presence of an outlier.

- If one of the standards returns bias low or bias high response on all of the analytes, the 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, drop the 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. If ICV is non-compliant, consider the following to correct the problem:

- If all analytes are bias low or bias high, the amount of standard injected could be inaccurate. Prepare another standard and re-analyze the ICV.
- If standard degradation is observed, prepare from a new standard lot and re-analyze the ICV.
- If the problem persists, perform instrument troubleshooting. Check leaks, check gas flow rates and refer to GC troubleshooting manual to correct the problem. Repeat the initial calibration and ICV.

12.2.3. If DCC is non-compliant, consider the suggestions in Sec. 12.3.2 to correct the problem:

12.2.4. If suggested corrective actions do not correct the problem, consult with the Supervisor for further advise prior to performing a new ICAL.

12.3. **Instrument Blank**

12.3.1. When instrument blank is non-compliant, fill a new tedlar bag with helium, clean the injection syringe and analyze the helium by direct injection. If the problem persists, either the injection port is contaminated or the helium source is contaminated. Consider the following suggestions to correct the problem.

- Check the helium source to make sure that the helium grade is ultra-high purity.
- Change the GC injector septa.
- Cut the column.

12.4. **Method Blank**

12.4.1. If method blank is non-compliant, consider the following suggestions to investigate the source of the problem.

- Check for possible contamination from the reagent water. If contamination is due to reagent water, change the filter at the source.
- Check for possible contamination from the vial.

- Analyze the vial septa. Cut the septa in half and place in a new vial. Add 2 drops of HCl and fill the vial with reagent water. Let it stand for at least 1 hour. Analyze like a regual sample.
- If contamination is from the vial, use another lot or vial manufacturer.

- If the analyte found in the method blank is not detected in any of the field samples, consult the Supervisor and the PM if the result can be reported. Otherwise, re-extract and re-analyze the method blank prior to sample analysis.

12.5. **Laboratory Control Sample**

12.5.1. If LCS/LCSD recovery fails, check that spike amount is correct and proper injection was achieved. Correct the problem and then re-extract and reanalyze the LCS and all associated samples.

12.6. **Matrix Spike**

12.6.1. If MS/MSD recovery fails, consider the following suggestions:

- Review the chromatogram and check if matrix interference is observed. Discuss in the case narrative.
- Check that the spike amount is correct and proper injection was achieved. Correct the problem, re-extract and reanalyze.

12.7. For insufficient amount of sample, note it in the sample preparation log, initiate an NCR, inform the PM immediately and discuss it in the case narrative.

12.7.1. When sample chemical preservation is insufficient (samples labeled preserved but the pH is >2), note it in the analytical run log comment section and discuss it in the case narrative.

12.8. **Method QC**

12.8.1. If LOD and LOQ verifications failed to meet the acceptance criteria, consider instrument maintenance prior to re-analyzing LOD/LOQ verification. Otherwise, perform new detection limit study.

12.8.2. If retention time has significantly shifted, check the carrier gas flow or check for leaks. Perform RTW study and apply as necessary.

12.9. **Non-Conformance Report (NCR)**

12.9.1. Initiate an NCR when the following circumstances occur:

- Anomalies other than specified in Appendix 1 is observed.
- Sample is out of technical holding time.

12.9.2. Refer to EMAX-QA08 for Non-Conformance Report details.

13.0 POLLUTION PREVENTION

13.1. Observe all necessary precautions to avoid spillage of solvent that may go to the wastewater drains.

13.2. To prevent internal pollution, sample extraction and analytical standard preparation must be conducted in a properly functioning fumehood.

14.0 WASTE MANAGEMENT

14.1. No samples should be dumped into 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 the analytical process in properly labeled satellite waste containers for proper collection.

15.0 SUPPLEMENTARY NOTES

15.1. A study of 40mL vials supplied by VWR International was conducted to establish the sample amount used for analysis. It was established that the sample amount is 39 mL. In the event that vials are purchased from another supplier, a similar study shall be conducted to establish the sample amount used in this analysis.

15.2. **Definition of Terms**

15.2.1. 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 sample 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.

15.2.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.2.3. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.2.4. Instrument Blank – is target-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.2.5. 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

15.2.6. Lab Control Duplicate Sample (LCD) – is a replicate of LCS analyzed to monitor precision when MS/MSD sample is not analyzed.

15.2.7. Matrix – is a component or form of a sample.

15.2.8. 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.

15.2.9. Matrix Spike Duplicate – is a replicate of MS analyzed to monitor precision or recovery.

15.2.10. Method Blank – is a target-analyte-free sample subjected tot he entire sample preparation and/or analytical to monitor contamination.

15.2.11. 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.

15.2.12. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.2.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.3. **Application of EMAX QC Procedures**

15.3.1. The procedures and QC criteria summarized in this SOP shall be applied to all samples unless otherwise other directive is specified by the project requirements.

15.4. **Department of Defense (DoD) and Department of Energy (DOE) Projects**

15.4.1. 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. DH Kampbell, JJ Wilson and S.A. Vandegrift. "Dissolved Oxygen and Methane in Water by a GC Head space Equilibration Technique". International Journal Environ Analytical Chem., Vol. 30, pp. 249-257, 1989

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 ICAL Summary

17.1.4. Figure 4 Typical Calibration Verification Summary

17.1.5. Figure 5 Typical Sample Result Summary

17.1.6. Figure 6 Typical LCS Summary Report

17.1.7. Figure 7 Typical Matrix Spike Summary Report

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.	RSK175FS	Sample Preparation Log
17.3.2.	RSK175FA	Analysis Run Log
17.3.3.	RSK175FM	Instrument Maintenance Log

Figure 1: **PEAK EVALUATION TECHNIQUE**

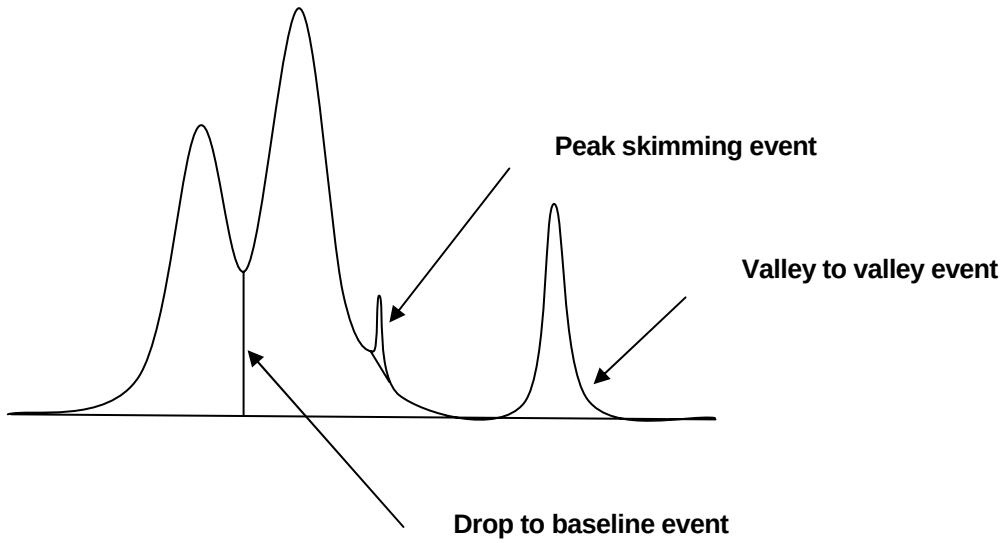


Figure 2: **TYPICAL CHROMATOGRAM**

EPA RSK-175 by GC/FID
EMAX Analytical Laboratories, Inc.

File : c:\ezc_9_~1\16chrom\fa06\fa06.008
Method : c:\ezchrom\method\dg10a06.met
Sample ID : IDG10A0601
Acquired : Jan 06, 2016 13:10:23
Printed : Apr 04, 2016 14:20:20
User : SCerva

Channel A Results

#	Peak Name	Ret. Time(Min)	Area	Ave. CF	ESTD Conc.(ppb)
1	Methane	1.058	472873.0	34639.0	13.65
2	Acetylene	2.233	80669.0	3585.6	22.50
3	Ethylene	2.650	493071.0	20628.8	23.90
4	Ethane	3.075	801299.0	30750.7	26.06
5	Propane	5.817	1199485.0	31353.2	38.26

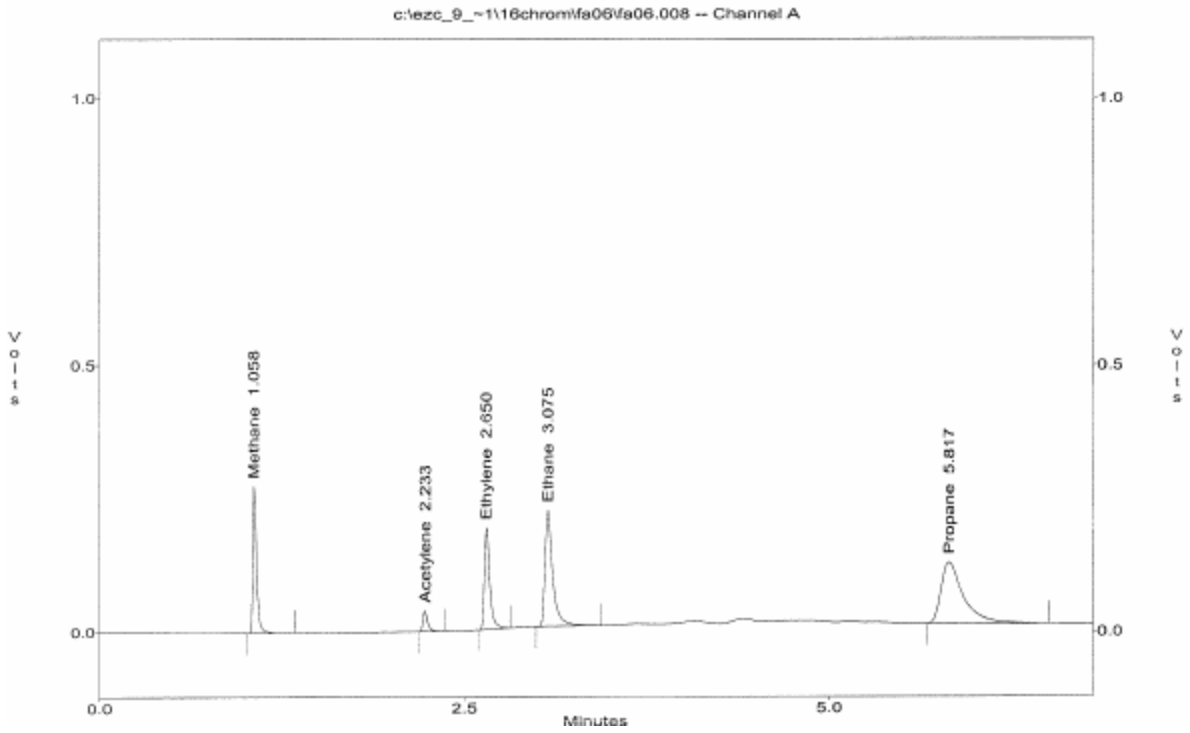


Figure 3: **TYPICAL ICAL SUMMARY**

INITIAL CALIBRATION
DISSOLVED GASES

Lab Name : EMAX Inc
Instrument ID : GCT010
GC Column : CARBOXEN 1006PLOT
Column size ID : 30MX0.53MM
LFID & Datetime: FA06002A 01/06/16 11:20
LFID & Datetime: FA06003A 01/06/16 11:35
LFID & Datetime: FA06004A 01/06/16 11:52
LFID & Datetime: FA06005A 01/06/16 12:04
LFID & Datetime: FA06006A 01/06/16 12:18
LFID & Datetime: FA06007A 01/06/16 12:32
CONC UNIT: ppb

COMPOUND	CONC X	CALIBRATION FACTORS (AREA)/UNIT 2.00X	10.00X	25.00X	40.00X	75.00X	100.00X	MEAN	%RSD
Methane	0.34	42297	35106	32366	33225	32782	32059	34639.0	11.3
Acetylene	0.55	3769	3725	3442	3537	3560	3481	3585.6	3.7
Ethylene	0.59	21531	20804	20079	20531	20676	20152	20628.8	2.6
Ethane	0.64	31856	31130	29747	30592	30883	30296	30750.7	2.4
Propane	0.93	32351	31144	30662	31390	31481	31091	31353.2	1.8

DG10A06.MET

INITIAL CALIBRATION
DISSOLVED GASES

Lab Name : EMAX Inc
Instrument ID : GCT010
GC Column : CARBOXEN 1006PLOT
Column size ID : 30MX0.53MM
LFID & Datetime: FA06002A 01/06/16 11:20
LFID & Datetime: FA06003A 01/06/16 11:35
LFID & Datetime: FA06004A 01/06/16 11:52
LFID & Datetime: FA06005A 01/06/16 12:04
LFID & Datetime: FA06006A 01/06/16 12:18
LFID & Datetime: FA06007A 01/06/16 12:32

COMPOUND	RT OF STANDARDS (MIN) 2.0X	10.0X	25.0X	40.0X	75.0X	100.0X	MEAN RT	RT WINDOW FROM	TO	RTWINDOW WIDTH
Methane	1.058	1.058	1.067	1.058	1.058	1.067	1.061	1.035	1.087	0.026
Acetylene	2.225	2.233	2.233	2.208	2.225	2.250	2.229	2.176	2.282	0.053
Ethylene	2.642	2.650	2.650	2.650	2.650	2.667	2.651	2.613	2.690	0.038
Ethane	3.067	3.075	3.075	3.075	3.075	3.092	3.076	3.038	3.115	0.038
Propane	5.808	5.817	5.817	5.817	5.817	5.842	5.820	5.770	5.870	0.050

DG10A06.MET

Figure 4: **TYPICAL CALIBRATION VERIFICATION SUMMARY**

INITIAL CALIBRATION VERIFICATION
DISSOLVED GASES

Lab Name : EMAX Inc
Instrument ID : GCT01D
GC Column : CARBOXEN 1006PLOT
Column size ID : 30MX0.53MM
Mid Conc Init LFID & Datetime: FA06005A 01/06/2016 12:04
Conc Cont LFID & Datetime: FA06008A 01/06/2016 13:10
CONC UNIT : ppb

COMPOUND	RT MINUTES	RT WINDOW FROM	RT WINDOW TO	TRUE CONC	AVERAGE CF	RESULT AREA	RESULT CONC	%D	QL	%D LIMITS
Methane	1.058	1.032	1.084	13.6	34639.0	472873	13.65	1		15
Acetylene	2.233	2.180	2.286	22.0	3585.6	80669	22.50	2		15
Ethylene	2.650	2.612	2.688	23.7	20628.8	493071	23.90	1		15
Ethane	3.075	3.037	3.113	25.4	30750.7	801299	26.06	3		15
Propane	5.817	5.767	5.867	37.3	31353.2	1199485	38.26	3		15

DG10A06.MET

Figure 5: **TYPICAL SAMPLE RESULT SUMMARY**

RSK-175
DISSOLVED GASES

Client	: XYZ, INC.	Date Collected:	01/06/16
Project	: CLEAN WATER	Date Received:	01/07/16
Batch No.	: 16A084	Date Extracted:	01/12/16 09:10
Sample ID:	SD024-24-MW-8A-2(1Q16)	Date Analyzed:	01/12/16 10:36
Lab Samp ID:	A084-02	Dilution Factor:	1
Lab File ID:	FA12006A	Matrix :	WATER
Ext Btch ID:	DGA005W	% Moisture :	NA
Calib. Ref.:	FA12002A	Instrument ID :	GCT010

PARAMETERS	RESULTS (ug/L)	LOQ (ug/L)	LOD (ug/L)
ACETYLENE	ND	2.0	0.55
ETHANE	6.3	2.0	0.64
ETHENE	9.3	2.0	0.60
METHANE	3.3	2.0	0.34
PROPANE	ND	2.0	0.90

Figure 6: **TYPICAL LCS SUMMARY**

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 16A084
METHOD: RSK-175

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	MBLK1W				
LAB SAMP ID:	DGA005WB	DGA005WL	DGA005WC		
LAB FILE ID:	FA12003A	FA12004A	FA12005A		
DATE EXTRACTED:	01/12/1609:10	01/12/1609:10	01/12/1609:10	DATE COLLECTED:	NA
DATE ANALYZED:	01/12/1609:54	01/12/1610:05	01/12/1610:24	DATE RECEIVED:	01/12/16
PREP. BATCH:	DGA005W	DGA005W	DGA005W		
CALIB. REF:	FA12002A	FA12002A	FA12002A		

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 (%)
Acetylene	ND	22.0	24.2	110	22.0	24.6	112	2	60-140	30
Ethane	ND	25.4	24.9	98	25.4	22.5	89	10	74-131	20
Ethene	ND	23.7	22.3	94	23.7	20.5	87	8	72-133	20
Methane	ND	13.6	12.7	93	13.6	11.7	86	8	73-125	20
Propane	ND	37.3	38.0	102	37.3	38.4	103	2	50-140	30

Figure 7: **TYPICAL MATRIX SPIKE SUMMARY REPORT**

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER
BATCH NO.: 16A084
METHOD: RSK-175

MATRIX:	WATER			% MOISTURE:	NA
DILUTION FACTOR:	1	1	1		
SAMPLE ID:	SD024-24-MW-8A-2(1Q16)				
LAB SAMP ID:	A084-02	A084-02M	A084-02S		
LAB FILE ID:	FA12006A	FA12007A	FA12008A		
DATE EXTRACTED:	01/12/1609:10	01/12/1609:10	01/12/1609:10	DATE COLLECTED:	01/06/16
DATE ANALYZED:	01/12/1610:36	01/12/1611:28	01/12/1611:47	DATE RECEIVED:	01/07/16
PREP. BATCH:	DGA005W	DGA005W	DGA005W		
CALIB. REF:	FA12002A	FA12002A	FA12002A		

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 (%)
Acetylene	ND	22.0	20.8	95	22.0	19.4	88	7	60-140	30
Ethane	6.3	25.4	25.6	76	25.4	25.0	74	2	74-131	20
Ethene	9.3	23.7	27.1	75	23.7	26.5	73	2	72-133	20
Methane	3.3	13.6	15.2	88	13.6	14.7	84	3	73-125	20
Propane	ND	37.3	35.8	96	37.3	34.7	93	3	50-140	30

Figure 8: **TYPICAL CASE NARRATIVE**

CASE NARRATIVE

Client : XYZ, INC.

Project: CLEAN WATER

SDG : 16A084

METHOD RSK-175
DISSOLVED GASES

A total of eleven (11) water samples were received on 01/07/16 to be analyzed for Dissolved Gases in accordance with Method RSK-175 and project specific requirements.

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 prepared and analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed. DGA005WB - result was compliant to project requirement. Refer to sample result summary form for details.

Lab Control Sample
Lab control sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of LCS/LCD was analyzed. All Analytes in DGA005WL/DGA005WC were within LCS limits. Refer to LCS summary form for details.

Matrix QC Sample
Matrix spike sample was prepared and analyzed at a frequency required by the project. For this SDG, one (1) set of MS/MSD was analyzed. All analytes were in control. Refer to Matrix QC summary form for details.

Sample Analysis
Samples were analyzed according to prescribed analytical procedures. Results were evaluated in accordance to project requirements. For this SDG, all quality control requirements were met with the exception of those that were discussed within the associated QC parameter.

Appendix 1: **SUMMARY OF QUALITY CONTROL PROCEDURES**

QC PROCEDURE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	1st Rvw	2nd Rvw
Five-point initial calibration for all analytes	Initially; as needed	% RSD for all analytes ≤ 20%	Correct the problem then repeat initial calibration		
Initial Calibration verification - ICV (Second-source)	After initial calibration	All analytes within ±25% of expected value	Correct the problem then repeat initial calibration		
Calibration verification	Bracket every 12 hours and at the end of the analytical run.	All analytes within ±15% 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 (≤ 20 samples per matrix)	No analytes detected ≥ ½ LOQ	If sample results are ND or sample result >10X than the contamination level, consult with the PM if results are reportable. Otherwise, re-extract and reanalyze method blank and all samples processed with the contaminated blank. If re-extraction/re-analysis is not possible, apply B to specific analyte(s) on all associated.		
LCS	As required by the project. One LCS per preparation batch (≤ 20 samples per matrix)	In the absence of project QC Limits use EMAX QC limits	Re-extract and reanalyze the LCS and all associated samples		
MS/MSD	As required by the project.	In the absence of project QC Limits use EMAX QC limits	If chromatogram exhibits matrix interference narrate observation in the case narrative. Otherwise, check that spike value added is appropriately accounted. If anomaly is suspected, re-extract and re-analyze the matrix spike samples.		
Comments: Refer to PSR for Flagging Criteria.			Reviewed by:		
			Date:		

Appendix 2: **DEMONSTRATION OF CAPABILITY**

DEMONSTRATION OF CAPABILITY
METHOD: RSK175

SOP: EMAX-RSK175 Rev. 2
Conc Unit: µg/L
Sample Amount(ml): 39
Extract Amount (mL): 4

Analysis date: 1/6/2016
Analyzed by: S. Cervantes

PARAMETER	FA06011	FA06012	FA06013	FA06014	TV	Ave. Conc.	Ave. %Rec	SD	RSD (%)	QC Criteria	COMMENTS
	DGA001WL	DGA001WC	DGA002WL	DGA002WC							
Methane	13.27	12.98	12.40	12.20	13.56	12.71	94	0.50	4	70 - 130	PASSED
Acetylene	22.38	22.46	21.56	21.82	22.00	22.06	100	0.44	2	30 - 160	PASSED
Ethylene	23.52	23.46	22.56	22.19	23.73	22.93	97	0.66	3	30 - 160	PASSED
Ethane	25.18	24.58	25.47	25.03	25.42	25.07	99	0.37	1	70 - 130	PASSED
Propane	37.07	36.40	34.39	33.79	37.28	35.41	95	1.57	4	70 - 130	PASSED

SOP: EMAX-RSK175 Rev. 2
Conc Unit µg/L
Sample Amount(g): 39
Volume Extracted(ml): 4

Date Analyzed: 01/11/16
Analyzed by: S. Cervantes

PARAMETER	CSA013	CSA014	CSA015	CSA016	TV	Ave. Conc.	Ave. % Rec.	SD	RSD	QC Criteria	Comments
	CA13A01L	CA13A01C	CA13A02L	CA13A02C							
Carbon Dioxide	9261	9244	9232	9309	9300	9262	100	34	0	60 - 130	Passed

RSK175FA: **ANALYTICAL RUN LOG**

EMAX LABORATORIES, INC.

ANALYTICAL RUN LOG
for
DISSOLVED GAS

Page 1

Note: For samples and relevant QCs/Standards analyzed, refer to attached analytical sequence.

Comments:

Book #: A10-037

Instrument No.: 10

Analytical Sequence:

Method File:

Analytical Batch:

SOP #	Rev. #
☐ EMAX-RSK175	3
☐ EMAX-D1945	0
☐ EMAX-	
☐ EMAX-	

STANDARDS ID		Conc (µg/L)
Temperature (°C)		
Data File		

ELECTRONIC DATA ARCHIVAL	
Location	Date
Labbkup/Ezchrom/EZC_9_DGAS	

SYRINGES
☐ 100 µL - MSF-01-02-01
☐ 100 µL - MSF-01-02-02
☐ 100 µL - MSF-01-02-20
☐ 10 µL - MSF-01-02-05
☐

Analyzed By:

Date:

Disposed By:

Date Disposed:

RSK175FS: **SAMPLE PREPARATION LOG**

EMAX LABORATORIES, INC.

Page 1

EXTRACTION LOG FOR DISSOLVED GAS

SOP ☐ EMAX-RSK175 Rev. 3 ☐ CO2 ☐ Room Temp. (°C): Book #: EDG-040

Start Date: Time: End Date: Time:

PREPARATION BATCH# _____

Sample Prep ID	Lab Sample ID	Sample Amount (ml)	Extract Volume (ml)	pH (< 2)	Notes
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					

Standards	ID	Amount Added (ul)
LCS/MS		

Reagent	Source
H_2O	
He	
Thermometer ID #:	
Tedlar Bag Lot#:	
pH Strips:	
Syringes	
☐ 10 mL - MSF-01-01-24	
☐ 5 mL - MSF-01-01-25	
☐	

Comments: _____

Prepared By: _____

Standard Added By: _____

RSK175FM: **INSTRUMENT MAINTENANCE LOG**

EMAX LABORATORIES, INC.

Page 1

INSTRUMENT MAINTENANCE LOG

Instrument No: 10 Month Year: 201_ Book # M10-006

DATE							
Computer Date/Time							
Printer Paper							
GC Keyboard Check							
Check Temperature Program							
Column Head Pressure							
Carrier Source Pressure*							
Bake GC							
H2 Generator 40-50 psi							
Instrument Blank							
Analyst's Initial							
Notes: * To insure proper operation, make sure that carrier source pressure is at least 20 psi greater than the column head pressure.							
Trouble Shooting							
Problem(s) Encountered							
Return-to-Control (Analyst Initial)							

Code	Problems Encountered	Code	Corrective Action

Title: Determination of Volatile Sulfur Compounds from Stationary Sources

[Methods EPA 15 & 16 and CARB 15 & 16]

Approvals (Signature/Date):

Koroush Vaziri Department Manager	6/9/16 Date	Joe Schairer Health & Safety Manager / Coordinator	6/6/16 Date
Lisa Stafford Quality Assurance Manager	6/27/16 Date	Crystal Pollock Laboratory Director	6/27/16 Date

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the gas chromatographic analysis of air or vapor samples for volatile sulfur-containing compounds using a Pulsed Flame Photometric Detector (PFPD). This SOP is based on EPA and CARB Methods 15 and 16. Other volatile sulfur-containing compounds may be analyzed using this method, depending on vapor pressure, compound reactivity, and project-required data quality objectives (DQOs).

1.2. Analytes, Matrix, and Reporting Limits (RLs)

1.2.1. Target analytes and RLs are listed in Attachment 1. Note that RLs are subject to change based on annual method detection limit (MDL) studies.

1.2.2. Applicable matrices – stationary source air, landfill gas

1.3. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air or vapor sample is introduced into a gas chromatographic system via a Sulfinert-treated stainless steel sample loop. Positive pressure is used to push the sample through the loop. Once the loop has equilibrated at ambient pressure, the gas chromatography (GC) carrier gas is diverted through the sample loop and the sample is swept onto a GC column. The sample is separated by a temperature-programmed capillary column. Detection occurs via a PFPD. Target analytes are identified by retention time (RT). Concentrations of target analytes are determined using external standard calibration technique.

2.2. Samples collected in air sample bags are loaded with a gas-tight syringe. Samples collected in SilcoCans, received at negative or ambient pressure, are first pressurized with Ultra High Purity Nitrogen (UHP N_2) prior to analysis and are connected directly to the sample line. Sample dilutions are performed by serial dilution methodologies using clean air sample bags and gas-tight syringes.

3. DEFINITIONS

3.1. Note that *"must"* and *"shall"* in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (e.g., SKC or ESS).

3.3. Part per million volume to volume (ppmv or ppm v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a million parts of gaseous volume of sample.

Note: This reporting unit is NOT equivalent to the common ppm unit used in soil or water analysis.

3.4. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most volatile organic compounds (VOCs). This type of canister is also called a Restek Sulfinert-treated (or Restek Siltek-treated) canister that is suitable for collecting samples containing sulfur compounds.

3.5. Standard molar volume is defined as 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.6. Standard pressure is defined as 1.0 atmosphere, 14.6 pounds per square inch absolute (psia), 0 inches of mercury, and 0 pounds per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum.

3.7. SUMMA canister: A spherical stainless steel container, of which the interior has been specially treated by a process (SUMMA passivation), that renders all surfaces inert to VOCs.

3.8. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in the SilcoCan. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All units used to express vacuum or pressure are converted to psia.

3.9. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.10. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. RTs must be carefully evaluated to eliminate false positives. Peak shape is carefully evaluated to determine if co-elution is apparent.

4.2. Data is carefully evaluated for carry-over and sample is re-analyzed if carry-over is suspected. Contamination from carry-over may occur when a lower concentration sample is analyzed after a relatively higher concentration sample.

4.3. Chromatograms must be carefully evaluated for baseline anomalies that may indicate the presence of quenching. Quenching results when the carbon monoxide produced by

the combustion of large amounts of hydrocarbons react with the sulfur atoms from a co-eluting sulfur species of interest. If quenching is suspected, then sample re-analysis at a different dilution is required to confirm the concentration of the sulfur analyte of interest.

4.4. TestAmerica Sacramento considers Restek Sulfinert-treated SilcoCan and canister valve be the only acceptable canister container for sulfur analysis. Use of other canister types (unless equivalent to SilcoCan), may affect the recovery of the sulfur compounds. SUMMA canisters have been shown to yield low or zero recovery when filled with a sulfur compound standard at the laboratory.

4.5. SilcoCans used in the laboratory to prepare sulfur standards must be plugged only with Restek Sulfinert-treated plugs, when available. Use of other types of plugs (unless equivalent to the Restek type) may affect the recovery of the sulfur compounds.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous.

Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

Danger: Hydrogen (H_2) is a flammable high pressure gas. It can form explosive mixtures with air. It may ignite if the valve is opened to air.

5.1.2. When pressurizing SilcoCans, safety glasses and a face shield must be worn. If pressure from the SilcoCan must be released during this process, a face shield must also be worn.

5.1.2.1. SilcoCans must never be pressurized over 40 psig.

5.1.3. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators or connecting/disconnecting supply lines.

5.1.4. Air sample bags must not be pressurized, as seam splitting will occur.

5.1.5. Some sample bags may smell heavily of sulfur. These should be placed in a sealed Ziploc bag until used, and ;may be kept in a fume hood.

5.1.6. The preparation of standards and reagents will be conducted in a fume hood with the sash set at the level indicated on the side of the hood.

5.1.6.1. Sulfur standard cylinders under 30” in height should be strapped inside the fume hood during standard preparation. The taller sulfur cylinders, over 4’ in height, should be strapped in front of the fume hood. The regulator outlet of these taller cylinders should be directed into the fume hood while standard is being released from the cylinder.

5.1.7. In order to prevent contamination of the laboratory air by the samples, the vent line must be connected to the system outlet and the fume hood must be turned on.

5.1.8. The GC oven contains elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the unit. Temperature appropriate gloves must be worn when working with hot or cold items.

5.1.9. Due to high voltage risk, power to the GC must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Carbon Disulfide	Health Flammable	10ppm ACGIH TLV 20ppm OSHA PEL	Reproductive, Mutagen Kidney, Liver, Heart damage. Flammable at 1.3%-50% in air
Ethanethiol	Health Flammable	0.5ppm TLV-TWA 10ppm OSHA PEL	Nervous system damage, fatal. Flammable at 2.8%-18.2% in air Can form explosive mixtures with air
Helium	Simple asphyxiant	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrogen	Flammable, Simple asphyxiant	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Methanethiol	Asphyxiant Health Flammable	0.5ppm TLV-TWA 10ppm STEL	Liver and Kidney damage Pulmonary edema Flammable at 3.9%-21.8% in air
Nitrogen	Simple asphyxiant	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Agilent 7890A (or similar) GC equipped with PFPD, capable of sub-ambient temperature programming and automated multiple valve control

6.1.2. Automated data system capable of archiving instrument runs (ChemStation or equivalent)

6.2. Supplies

6.2.1. Restek Siltek-treated (Restek Sulfinert) stainless steel tubing, Swagelok fittings, Valco valves (custom configuration from Lotus Consulting)

6.2.2. Agilent DB-1, 60 m x 0.320 mm x 5 um capillary column

6.2.3. Restek Sulfinert treated SILCO canister with treated canister valve

6.2.4. Air Sample Bags, 3-L or 1-L (SKC, ESS, or equivalent).

6.2.5. Gas-tight syringes, various sizes 1.0mL to 1-L.

6.2.6. Transducer and process meter capable of measuring 0 psia to 50 psia.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP Helium (He) for carrier gas

7.1.2. UHP H2 for detector combustion

7.1.3. Zero-grade air for detector combustion

7.1.4. UHP N2 for sample preparation and standard preparation

7.2. Stock Standards

Gas calibration standards in cylinders containing compatible target compounds at convenient levels. These standards are certified by the supplier (e.g., Praxair and Air Liquids).

7.3. Working Standards

Working standards are prepared at appropriate levels by making dilutions from the certified gas cylinders in UHP N2. Dilutions are performed using de-activated glass syringes or de-activated pressure transmitter/gauge and air sample bags or Restek Sulfinert-treated canisters (or equivalent). See Attachment 3 for the nominal concentration levels used in the initial calibration (ICAL).

7.3.1. Working Gas Standard Prepared in Restek Sulfinert-treated (or equivalent) Canisters – To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated Restek Sulfinert-treated (or equivalent) canister. The aliquot is metered in by measuring the vacuum of the Restek Sulfinert-treated (or equivalent) canister as the aliquot is being transferred. The Restek Sulfinert-treated (or equivalent) canister is pressurized with UHP N2 and is then allowed to equilibrate. The ratio of the final pressure and the pressure transferred is the dilution factor (DF).

7.3.2. Working Gas Standard Prepared in Air Sample Bags – To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.3. To calculate the concentration of the standard prepared:

$$ppmv = C_{ps} \times P_i / P_f$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL (if using an air sample bag)

P_f = final pressure of passivated canister in psia or final volume in mL (if using an air sample bag)

7.4. The preparation of each standard is recorded in the Laboratory Information Management System (LIMS).

7.5. Expiration dates for standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Expiration dates must be documented on the gas cylinders.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
SilcoCan	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
SilcoCan	2000 mL	None	72 hours	Advisory – Active Soil Gas Investigations, January 28, 2003 (DTSC and LARWQCB)
Air Sample Bag	500 mL	None	72 Hours	N/A

8.2. Samples should be collected in either air sample bags or Restek Sulfinert-treated (or equivalent) SilcoCans equipped with treated valves.

8.2.1. If a SilcoCan (or equivalent) is used, a treated pressure gauge or treated pressure transmitter is used to record the absolute pressure of the SilcoCan sample upon receipt and after pressurization (if required) at the laboratory.

8.2.2. The pressure should also be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. Preservatives are not required by the method.

8.6. Samples are stored at room temperature.

9. QUALITY CONTROL

9.1. Laboratory Control Samples: LCS's are laboratory-generated samples used to monitor the laboratory's day-to-day performance. The LCS is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS is prepared from a source independent of the calibration standards.

9.2. Method Blank: The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. The method blank, consisting of all reagents added to the samples, is analyzed with each batch and is processed in the same manner as samples.

9.3. Batch
A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Continuing Calibration Verification (CCV), a Laboratory Control Sample (LCS), a Method Blank, and a closing CCV, but they do not count towards the maximum 20 samples in a batch.

9.3.1. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.3.2. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch count.

9.3.3. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors (RFs) must be in effect throughout the analysis.

9.3.4. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.4. Laboratory Control Sample

For each batch of samples, an LCS must be analyzed after the calibration standard and before the method blank and samples. The LCS, obtained from a second-source, is spiked with all the target analytes. The LCS, as quantitated against the ICAL curve, must meet laboratory derived control limits for each target analyte. The concentration of the spiked analytes shall be at or below the midpoint of the calibration curve.

9.4.1. Refer to the QC Program document (WS-PQA-003) for details on the requirements for LCS/LCSD composition. Sample/sample duplicate analyses may be used in place of LCS/LCSD analyses for this method.

9.4.2. If any analyte is outside established control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the batch. Refer also to the troubleshooting guidelines in Section 11.5.

9.4.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.4.4. All data reported with out of control sample/sample duplicate values will be flagged by the LIMS. Analysts should also file a nonconformance memo (NCM) within the LIMS detailing why the data is reported, and any corrective actions performed.

9.4.5. Corrective actions to occur before batch reanalysis:

9.4.5.1. Evaluate the analytical run for errors and anomalies. Re-analyze the LCS.

9.4.5.2. Consult the troubleshooting guidelines in Section 11.5. Evaluate the instrument status and perform maintenance.

9.4.5.3. Re-analyze the continuing calibration verification (CCV) standard and LCS, or recalibrate.

9.4.6. Current LCS control limits are stored in the LIMS. Control limits are subject to change based on periodic evaluation of LCS control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.5. Method Blank

For each batch, an acceptable method blank must be analyzed. The method blank is analyzed after the calibration standards and LCS/LCSD and prior to client samples. The method blank, prepared in either a Restek Sulfinert-treated (or equivalent) canister or a clean air sample bag, will consist of UHP N2.

9.5.1. The method blank must not contain any target analyte of interest above the RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements). Otherwise, the Method Blank is further evaluated and corrective actions must be performed, as stated below. See troubleshooting guidelines in Section 11.5.

9.5.1.1. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.5.1.2. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.5.1.3. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.5.1.3.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager (PM) must record the client's decision in the NCM.

9.6. Sample Duplicate

9.6.1. Precision information may be gained through the analysis of a duplicate sample injection. The RPD for sample duplicates is evaluated for all positive detections greater than the reporting limit, and compared to the limits in the LIMS.

9.6.2. If the RPD exceeds the control limits, the data is evaluated to determine if laboratory error (poor injection technique, etc) is involved. If so, the error is corrected and the duplicates reanalyzed.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to CA-Q-P-003, "Calibration Curves & Selection of Calibration Points".

10.2. Initial Calibration

10.2.1. Instruments must be calibrated at initial setup and as needed thereafter, and at least annually.

10.2.2. Prior to sample analysis, an ICAL curve, consisting of a minimum of 6 points (for a second-order curve) at different concentrations, is analyzed to determine the working range of the analytical system for each analyte. The lowest calibration level for each analyte should be at or below its RL. The highest calibration level is considered to represent the upper range of the analytical system for the corresponding analyte.

10.2.2.1. The calibration curve is weighted by 1/Concentration^2 (often called $1/X^2$ weighting) to improve the accuracy at the low end of the curve.

10.2.2.2. The y-intercept fit (forced, ignore, or include) must be chosen such that it is within ±½ the RL for a given analyte. The best intercept fit is one that minimizes the %D of an analyte's lower calibration level from the curve.

10.2.2.3. The ICAL curve for each target analyte is considered acceptable if the coefficient of determination (r2) is ≥ 0.990.

10.2.3. If the r2 does not meet the required acceptance criterion, the corresponding curve is considered invalid and corrective action must be performed. See troubleshooting guidelines in Section 11.5.

10.2.4. A new ICAL is required when situations like, but not limited to, the following are encountered:

- After changes are made to the original ICAL instrument configuration
- After replacement of analytical columns
- After replacement of detectors

10.2.5. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.2.6. Initial Calibration Verification (ICV)

Each new multi-point calibration must be verified using a second-source standard. The second-source standard, as quantitated against the new ICAL curve, must have recoveries between 85% and 115% for each target analyte. If these criteria are not met, the following corrective actions must be performed:

- Rerun the second-source check standard.
- Re-prepare or acquire a new standard.
- Evaluate instrument conditions or perform maintenance or repair, if needed. Instrument maintenance or repair must be documented in the instrument maintenance logbook.
- Re-analyze a new ICAL.

10.2.7. An acceptable ICAL and ICV is documented using a GC Initial Calibration Curve Review Checklist (see Attachment 6). This checklist is submitted to the Department Manager, or designee, for second-level review and signature. Only after successful completion of first and second-level reviews may the analysis be reported from the new ICAL.

10.3. Continuing Calibration Verification

10.3.1. A batch may be started after an approved ICAL and ICV have been completed.

10.3.2. An opening CCV standard is analyzed at the onset of each batch, or every 24-hour shift, whichever comes first, to verify the linearity of the ICAL. The CCV will contain each target analyte of interest with a source different from that used in the ICV. The concentration of CCV shall be at or below the midpoint of the calibration curve. The system is considered in control if the %D between the response of the target analyte in the CCV and the response of the target analyte in the ICAL is ± 15.

10.3.3. If the CCV fails acceptance criteria, corrective actions must be performed. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10e, pages 216 and 217 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92), if routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated. See troubleshooting guidelines in Section 11.5.

10.3.3.1. Repeated failure of the same target analyte is an indication of a <u>systematic deficiency</u> that must be corrected.

10.3.3.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the CCV after documenting and correcting the exact condition that caused the failure. Such failures may be due, but not limited, to poor standard preparation, low standard pressure, closed standard valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

10.3.3.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair, which must be documented in the instrument maintenance logbook. After maintenance or repair, the CCV (if this still meets the NELAC or TNI requirement stated in Section 10.3.3 above) may be re-analyzed. Otherwise, a new ICAL is required.

10.3.4. Following the last injected sample, the batch is closed with a passing CCV, which must also meet the same acceptance criteria as the opening CCV. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10c, page 216 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2c, page 92), acceptable opening and closing CCVs must bracket reportable samples.

10.3.4.1. If the closing CCV fails acceptance criteria, follow the corrective action procedures defined in Section 10.3.3.

10.3.4.2. If any target analyte in the closing CCV exceeds acceptance criterion and the target analyte was ND in the associated samples, no further corrective action is required. However, the nonconformance must be reported in an NCM.

10.3.4.3. If any sample result is reported from a batch where the closing CCV failed acceptance criteria, the CCV failure must be documented in an NCM. The NCM must indicate the reason why the sample results, in the best judgment of the analyst, are being reported under a failed closing CCV.

10.3.5. If sample analysis must be halted for more than an 8-hour hour period, an opening CCV must be analyzed prior to continuance of the batch, to ensure that instrument conditions have remained stable.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation

11.2.1. For air sample bag samples, the bag is checked for damage and is analyzed as received.

11.2.2. For Restek Sulfinert-treated (or equivalent) canister samples, the initial pressure is checked by attaching a treated vacuum/pressure gauge or treated pressure transmitter to the canister. The treated vacuum/pressure gauge or treated pressure transmitter must be rinsed before use with the pressurization gas (UHP N2) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the treated vacuum/pressure gauge or treated pressure transmitter attached, the Restek Sulfinert-treated (or equivalent) canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in the Canister Pressurization Logbook (see Attachment 7) and on the individual Canister Field Data Record (see example form in SOP LA-SRA-001).

11.2.3. If the canister pressure is increased, a dilution factor is calculated and applied to the raw results:

$$DF = \frac{Y_a}{X_a}$$

Where:

Xa = absolute initial canister pressure (before dilution, psia)

Ya = absolute final canister pressure (after dilution, psia)

11.3. Calibration

11.3.1. Before any instrument is used as a measurement device, the instrument

response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.3.2. Refer to Section 10 for details regarding instrument calibration.

11.4. Sample Analysis

11.4.1. The calibration standards and the sample QC are analyzed in the same manner as client samples. Before client samples can be analyzed, calibration standards and batch QA must be analyzed and evaluated.

11.4.2. Recommend instrument operating conditions are listed in Attachment 2.

Note: If, after instrument maintenance or repair, the parameters listed in Attachment 2 change, then the updated parameters must be listed in the instrument maintenance logbook. The appropriate QC is performed prior to analysis of samples to verify method performance.

11.4.3. The Sample List (also known as injection or run log) is constructed. The appropriate Autolink command and parameters in the data system should be included to facilitate data upload. The appropriate method and Sample List are activated by downloading to the analytical instrument.

11.4.4. The sample container is attached to the assigned sample port of the GC system or directly injected by syringe into the sample port for sulfur analysis. If the container is hooked up directly, the sample container valve is opened. Once the sample is flushed through the sample line, the analytical run is started.

11.4.4.1. The sample line is flushed with the sample for a period of time that guarantees proper rinsing. The analytical system will automatically adjust the sample loop to one atmosphere before injection.

11.4.4.2. After sample loop equilibration, a valve is actuated which diverts the flow of carrier gas through the sample loop and, hence, sweeps the contents of the sample loop onto the GC column. The GC column is initially at sub-ambient temperature to aid in focusing the lighter compounds.

11.5. Troubleshooting Guidelines

11.5.1. Low pressure in Restek Sulfinert-treated (or equivalent) canisters containing the standards or problems with carrier/detector gas supply: Always confirm

that adequate pressure remains and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.5.2. Low Response: Causes are typically leaking sample lines or valves and detector leaks. Check each sample line or valve in question by isolating it from the system (disconnect and cap or plug the ends) and then performing a leak test using a vacuum/pressure gauge and Restek Sulfinert-treated (or equivalent) canister at positive pressure. Leaking components will not hold pressure when the Restek Sulfinert-treated (or equivalent) canister is closed. Check the tightness of the jet retaining screws and the condition of the jet ferrule; replace if necessary.

11.5.3. Baseline Noise: Check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over.

11.5.4. Instrument Issues: If data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.6. Refer to Attachment 4 for a schedule of routine maintenance for the analytical instrument.

12. CALCULATIONS/DATA REDUCTION

12.1. After the analytical run has ended, a real-time review of the data is performed to determine if a successful analysis has been achieved. Peaks which exceed the upper calibration range and/or which exceed the signal output of the detector must be diluted and re-analyzed.

12.1.1. Sample dilution, when needed, is typically accomplished by transferring an aliquot of the sample into a tedlar air bag. For very large dilutions, the diluted sample can be diluted again into another air sample bag.

12.2. If the sample was analyzed at the proper dilution, the chromatogram/data is evaluated to determine if proper peak identification and integration was performed.

12.2.1. Qualitative Identification

A target analyte is identified by comparison of the sample analyte RT with that of the standards analyzed within the corresponding batch.

12.2.2. Quantitative Identification

12.2.2.1. When a target analyte has been identified, the quantitation of that target analyte will be based on its ICAL RF, the integrated area of the peak as performed by the instrument data system, and the DF. Refer to Section 4.3 regarding quenching.

12.2.2.2. The data system automatically quantitates the sample results. If a Restek Sulfinert-treated (or equivalent) canister sample was pressurized before analysis, the results must be multiplied by the DF (see Section 11.2.3).

12.2.2.3. If, in the best judgment of the analyst, the data system integration is incorrect, then the analyst may manually integrate the peak.

12.3. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.4. Calculations

12.4.1. Calculation for second-order Response Factor

$$y = ax^2 + bx + c$$

Where:

y = area response, uv-sec

x = analyte concentration, pomp

c = y-intercept

Solve the above equation for analyte concentration (quadratic formula):

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

12.4.2. Calculation for RPD

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ \ Values} X\ 100$$

12.4.3. Calculation for %D

$$\%D = \frac{\text{Expected - Result}}{\text{Expected}} \times 100$$

12.4.4. Calculation for %Rec

$$\%\text{Rec} = \frac{\text{Amount cpd recovered}}{\text{Amount cpd spiked}} \times 100\%$$

12.4.5. Reporting unit is typically ppmv. If results are to be reported in units of µg/L (also mg/m3), use the following equation:

$$\text{Result } (ug\ /\ L) = \frac{\text{Result } (ppmv\ ) \times (Molecular\quad Weight\ )}{24.45} X\,100$$

Note: 24.45 is the volume of ideal gas at 25°C and 1 atmosphere

µg/L = mg/m³

12.4.6. Calculation for Total Reduced Sulfur (TRS):

TRS = sum of the corrected concentrations of each calibrated sulfur compound

Note: The corrected concentrations of the disulfur analytes (carbon disulfide and dimethyl disulfide) are multiplied by two.

12.5. No conversion of the analytical results to standard conditions is made.

12.6. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of an appropriate review checklist is found in Attachments 6.

12.6.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.2. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood prior to discarding in facility garbage containers. They are damaged (e.g., a hole is drilled into the cylinders) so they cannot be reused.

15.3. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.4. Air sample bags are disposed when no longer needed. Disposal involves the slashing of the bags in a hood and allowing them to vent completely in a fume hood. Once vented, they are placed into a plastic Ziploc bag and put into the orange lab trash can.

When the can is full, or after no more than one year, it is taken to main waste for disposal.

16. REFERENCES/CROSS REFERENCES

16.1. EPA Method 15, Hydrogen Sulfide, Carbonyl Sulfide, and Carbon Disulfide (Promulgated). Posted 2/2000 on the US EPA Emissions Measurement Center - CFR Promulgated Test Methods (TM) website

16.2. EPA Method 16, Sulfur, Semicontinuous Determination (Promulgated). Posted 2/2000 on the US EPA Emissions Measurement Center - CFR Promulgated Test Methods (TM) website

16.3. California Air Resources Board (CARB) Method 16, Semicontinuous Determination of Sulfur Emissions from Stationary Sources, Adopted June 2, 1983, Amended July 1, 1999

16.4. California Air Resources Board (CARB) Method 15, Determination of Hydrogen Sulfide, Carbonyl Sulfide, and Carbon Disulfide Emissions from Stationary Sources, Adopted June 2, 1983, Amended July 1, 1999

16.5. EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd edition, January 1999

16.6. Advisory – Active Soil Gas Investigations, January 28, 2003 (DTSC and LARWQCB)

16.7. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.8. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.9. TestAmerica Sacramento QAM, current revision

16.10. TestAmerica Sacramento SOP WS-PQA-001, Quality Control Program, current revision

16.11. TestAmerica Costa Mesa SOP LA-QAS-002, Standards Preparation, Traceability, and Verification, current revision

16.12. TestAmerica Costa Mesa SOP LA-SRA-001, Sample Receiving, Login, and Internal Chain of Custody Procedures for Air Samples, current revision

16.13. TestAmerica Sacramento SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision

16.14. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.15. TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices, current revision

16.16. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.17. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

17. METHOD MODIFICATIONS

17.1. Method EPA 15 section 8.2.3 states that "a sample run is composed of 16 individual analysis (injects) performed over a period of not less than 3 hours or more than 6 hours". TestAmerica Sacramento's reportable sample analysis must occur within an analytical batch. An analytical batch may contain up to 20 client samples analyzed within a 24-hour period.

17.2. Methods CARB 15 and 16 specify flame photometric detection (FPD) of sulfur emissions. TestAmerica Sacramento uses a Pulsed Flame Photometric Detector (PFPD). The PFPD is considered to have improved selectivity and detection over the FPD. See RLs in Attachment 1.

17.3. Method CARB 15 Section 4.1 specifies precision requirement of three consecutive injections that do not vary by more than 15% of the mean. Method CARB 16 Section 4.1 specifies precision requirement of three consecutive injections that do not vary by more than 5% of the mean. TestAmerica Sacramento controls precision using a second-source LCS/LCSD pair for each batch of samples analyzed. The LCS/LCSD must fall within historically-determined accuracy and precision acceptance criteria.

17.4. Methods CARB 15 and 16 Section 4.2 specifies calibration drift "from the mean of three injections made at the beginning and end of any run or series of runs within a 24-hour period shall not exceed 5 percent." TestAmerica Sacramento uses a single injection of a CCV standard at the onset of each batch and at the close of each batch. The %D of the CCV relative to the multi-point calibration must be ±15. This is consistent with SW-846 GC methods utilized by the laboratory.

17.5. Methods CARB 15 and 16 Section 5.3.4.4 specifies that Teflon-lined valves, Teflon tubing, and Teflon fittings be used. TestAmerica Sacramento uses Restek's Sulfinert-treated sampling lines, fittings, and loops. TestAmerica Sacramento uses non-treated Valco valves since losses due to these valves have been shown to be insignificant at the RLs specified in Attachment 1.

17.6. Methods CARB 15 and 16 Section 6.5 specifies using permeation tubes for standard preparation. TestAmerica Sacramento prepares standards in air sample bags or Restek Sulfinert-treated sampling canisters (equipped with Restek Sulfinert-treated valves) from cylinder gas standards. The gas standards in cylinders are prepared and certified by an outside vendor.

17.7. Methods CARB 15 and 16 Sections 8.3 and 8.4 specify a three-point calibration curve, with each level analyzed in triplicate, spanning the linear range of the FPD. TestAmerica Sacramento' deviation from the methods' precision criteria was addressed in this SOP. TestAmerica Sacramento does not operate the PFPD in the square-root mode, and hence, a linear curve is not obtained. TestAmerica Sacramento uses a minimum of six calibration levels per analyte that establish the reporting range for each respective analyte. Curve fit is either second- or third-order per analyte.

17.8. Methods CARB 15 and 16 Sections 10.2 and 10.3 provide instruction on 24-hour partial recalibration standard analysis and acceptance criteria. TestAmerica Sacramento uses a single injection of a CCV standard at the onset of each batch and at the close of each batch. The %D of the CCV relative to the multi-point calibration must be ±15. This is consistent with SW-846 GC methods utilized by the laboratory

18. ATTACHMENTS

18.1. Attachment 1: Target Analytes and Reporting Limits

18.2. Attachment 2: Suggested Instrument Operating Parameters for Sulfur Analysis in Instrument GC7

18.3. Attachment 3: Nominal ICAL Concentrations

18.4. Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

19. REVISION HISTORY

19.1. WS-GCA-0019, Revision 1.2, Effective 06/28/2016.

19.1.1. Section 9.4.1 – Added sample/sample duplicate may be analyzed in place of LCS/LCSD for this method.

19.1.2. Updated RL's in Attachment 1.

19.1.3. Editorial changes

19.2. WS-GCA-0019, Revision 1.1, Effective 08/24/2015

19.2.1. Deleted Section 4.6 - Referenced pre-priming air bags with H_2S.

19.2.2. Section 7.3.2 – Removed reference to pre-primed air sample bag.

19.2.3. Section 9.4 – Replaced “must have recoveries between 75% and 125% for each target analyte” with “must meet laboratory derived control limits for each target analyte.”

19.2.4. Deleted Section 11.4.3 – Referenced pre-priming air bags with H2S.

19.2.5. Editorial changes.

19.3. WS-GCA-0019, Revision 1.0, Effective 7/24/2015

19.3.1. Updated Copyright statement (cover).

19.3.2. Section 7.2 – Updated vendors to Praxair and Air Liquids

19.3.3. Editorial changes

19.4. WS-GCA-0019, Revision 0.1, Effective 1/10/2014

19.4.1. Section 10.2.6, first sentence of the first paragraph, changed to read: “The second-source standard, as quantitated against the new ICAL curve, must have recoveries between 85% and 115% for each target analyte.”

19.4.2. Section 10.3.2, last sentence, changed to read: “The system is considered in control if the %D between the response of the target analyte in the CCV and the response of the target analyte in the ICAL is ±15.”

19.4.3. Section 17.4, changed to read: “Methods CARB 15 and 16 Section 4.2 specifies calibration drift “from the mean of three injections made at the beginning and end of any run or series of runs within a 24-hour period shall not exceed 5 percent.” TestAmerica Sacramento uses a single injection of a CCV standard at the onset of each batch and at the close of each batch. The %D of the CCV relative to the multi-point calibration must be ±15. This is consistent with SW-846 GC methods utilized by the laboratory.”

19.4.4. Section 17.8 changed to read: “Methods CARB 15 and 16 Sections 10.2 and 10.3 provide instruction on 24-hour partial recalibration standard analysis and acceptance criteria. TestAmerica Sacramento uses a single injection of a CCV standard at the onset of each batch and at the close of each batch. The %D of the CCV relative to the multi-point calibration must be ±15. This is consistent with SW-846 GC methods utilized by the laboratory.”

19.4.5. Removed attachment 4, renumbered attachment 5 as attachment 4.

19.5. WS-GCA-0019, Revision 0, Effective 04/19/2013

19.5.1. First version of this SOP.

Attachment 1: Target Analytes and Reporting Limits

Compound	RL, ppmv
Hydrogen sulfide	0.40
Carbonyl sulfide	0.10
Methanethiol (Methyl mercaptan)	0.30
Ethanethiol (Ethyl mercaptan)	0.40
Dimethyl sulfide	0.10
Carbon disulfide	0.20
Dimethyl disulfide	0.20

Attachment 2: Suggested Instrument Operating Parameters for Sulfur Analysis in Instrument ATGC4

Column Oven	
Initial Column Temperature	40
Initial Column Hold (min.)	5.0
Program 1 Temperature °C	-50
Program 1 Rate °C/min.	40.0
Program 1 Hold (min.)	1.00
Program 2 Temperature °C	4
Program 2 Rate °C/min.	30.0
Program 2 Hold (min.)	1.95
Program 3 Temperature °C	85
Program 3 Rate °C/min.	20.0
Program 3 Hold (min.)	2.45
Program 4 Temperature °C	180
Program 4 Rate °C/min.	20.0
Program 4 Hold (min.)	0.00

Valve Table	
Initial:	-1-2+4
0.01 min.:	+1+3+4
2.15 min.:	-1-2+4
2.45min.:	-1-3-4
3.80min.:	-1-3+4

Temperature Zones	
Rear Valve	150°C

Data Acquisition	
Bunch:	5Hz
Monitor:	3.2sec
Rear Scale:	10V

Rear PFPD Detector	
Temperature:	220°C
Square Root:	Off
PM Volts:	500
Gate Delay:	6.0msec
Gate Width:	20.0msec
Trigger Level:	200mv
Range:	10
AZ:	Yes
Air 1 Flow:	20ml/min
H2 Flow:	13.0ml/min
Air 2 Flow:	10.0ml/min

Front Injector	
EFC Type:	1
Column Flow:	8ml/min
Split:	ON
Ratio:	1

Note: Actual analytical parameters are determined from the daily evaluation of system performance.

Attachment 3: Nominal ICAL Concentrations

	ppmv					
Analyte	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
Hydrogen sulfide	0.1	0.2	0.5	0.8	1.0	2.0
Carbonyl sulfide	0.1	0.2	0.5	0.8	1.0	2.0
Methanethiol (Methyl mercaptan)	0.1	0.2	0.5	0.8	1.0	2.0
Ethanethiol (Ethyl mercaptan)	0.1	0.2	0.5	0.8	1.0	2.0
Dimethyl sulfide	0.1	0.2	0.5	0.8	1.0	2.0
Carbon disulfide	0.1	0.2	0.5	0.8	1.0	2.0
Dimethyl disulfide	0.1	0.2	0.5	0.8	1.0	2.0

Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.
As needed	Clip front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. All Detectors: Clean when baseline indicates contamination or when response is low. Autosamplers: Leak check system. Clean sample lines and valves. Replace gas supply. Bake column.

Title: Determination of Fixed Gases (Reformed Gases) in Air Samples using Gas Chromatography

[Methods ASTM D-1946 / EPA 3C / ASTM D-1945 / ASTM D-3588]

Approvals (Signature/Date):

Koroush Vaziri Department Manager 4/7/16 Date	Joe Schairer Health & Safety Manager / Coordinator 4/7/16 Date
Lisa Stafford Quality Assurance Manager 5/27/16 Date	Crystal Pollock Laboratory Director 5.31.16 Date

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the gas chromatographic analysis of air or vapor samples for fixed gas compounds using thermal conductivity detectors (Front TCD/Aux TCD).

1.2. This SOP is restricted for use by experienced analysts or those under the supervision of analysts experienced in sample preparation, use of gas chromatography (GC), and interpretation of chromatograms.

1.3. Analytes, Matrix, and Reporting Limits (RLs)

1.3.1. Target analytes and RLs are listed in Attachment 1. Note that RLs are subject to change based on annual LOQ/LOD studies.

1.3.2. Applicable matrices – ambient air, indoor air

1.4. This SOP also describes the calculations used to report Natural Gas composition (ASTM D1945) and "Heat Value and Relative Density of Gaseous Fuels" (ASTM D-3588). To calculate these values, results from Method TO-3 (WS-GCA-0018) are also required.

1.5. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air or vapor sample is injected with a gas-tight syringe into a sample loop or by directly connecting a canister to the sample line and onto HaySep and Mol Sieve columns. The system automatically adjusts the sample loop to one atmosphere before injection. Oxygen(O2), nitrogen (N2), and methane (CH4) are the first to pass through the HaySep and are then directed onto the Mol Sieve. After they have eluted onto the Mol Sieve, the Mol Sieve is isolated. Carbon dioxide (CO2) is next to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Carbon monoxide (CO) is last to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Ultra high purity (UHP) helium (He) is used as the carrier gas.

2.2. Hydrogen (H2) and Helium (He) may also be analyzed. UHP N2 is used as the carrier gas. After the elution of these compounds from the HaySep onto the Mol Sieve, the HaySep is back-flushed to vent. H2 and He elute from the Mol Sieve and are detected by the TCD.

3. DEFINITIONS

3.1. Note that "must" and "shall" in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volume, that is constructed of proprietary material (e.g., SKC or ESS).

3.3. Fixed gases: Fixed gases are a group of atmospheric gases which include N2, O2, CO2, CO, CH4, H2, and He.

3.4. Part per million volume to volume (ppmv or ppm v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a million part of gaseous volume of sample. One ppmv is equal to 1/10000 of a % v/v.

Note: This reporting unit is NOT equivalent to the common ppm unit used in soil or water analysis.

3.5. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-liter, 1.8-liter, 6-liter, or 15-liter volume.

3.5.1. SUMMA canister: A spherical stainless steel container, which interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to VOCs.

3.5.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.5.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek Corporation using a proprietary electropolishing process and extensively cleaned using an ultrasonic method that ensures a high-quality passivated surface that maintains the stability of VOCs during storage.

3.6. Percent by volume (% v or % v/v): Concentration expressed as percentage of gaseous (vapor) volume of pure target compound contained in a gaseous volume of sample; also known as mole percent.

3.7. Standard Dry Air: The laboratory bases its analysis/calculations on the following composition:

Nitrogen	78.1%
Oxygen	20.9%
Argon	0.918%
Carbon dioxide	0.034%
% Total	100.000%

3.8. Standard molar volume = 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.9. Standard pressure = 1.0 atmosphere or 14.6 pounds per square inch absolute (psia) or 0 inches of mercury or 0 pounds per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum.

3.10. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia.

3.11. Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.12. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. The analytical system is relatively free of interferences due to the multiple column configuration and limited number of similar compounds. Compounds of interest are well separated and there are no major baseline upsets to complicate correct peak integration.

4.2. Contamination may occur in the sampling systems if sample containers are not properly cleaned prior to use.

4.3. Air sample bags should also be shown to be free of contamination at levels one-half the RL for all target analytes.

4.4. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned and checked for leaks to ensure that the filling apparatus will not contaminate samples.

4.5. The potential for result bias due to carry-over exists. When carry-over is suspected, the sample must be re-analyzed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems

associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

Danger: H_2 is a flammable high pressure gas. It can form explosive mixtures with air. It may ignite if the valve is opened to air.

5.1.2. When pressurizing canisters, safety glasses must be worn. If pressure from a canister must be released during this process, a face shield must also be worn.

5.1.2.1. Passivated canisters should never be pressurized over 40 psig.

5.1.3. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators or disconnecting / connecting cylinders..

5.1.4. Air sample bags must not be pressurized, as seam splitting will occur.

5.1.5. Air sample bags may be used for standard preparation. These must be handled with care and must not be over-filled to prevent them from rupturing. Make sure the valves are closed tightly when not in use.

5.1.6. In order to prevent contamination of the laboratory air by the samples, the vent line must be connected to the system outlet and the fume hood must be turned on.

5.1.6.1. The effluents from the sample splitters for the GC must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.1.7. The GC oven contains elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the unit. Temperature appropriate gloves must be worn when working with hot or cold items.

5.1.8. Due to high voltage risk, power to the GC must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. There are no materials with a health rating of 3 or 4 used in this method **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methane	Flammable at 5% to 15% in air; Asphyxiate	PEL – Simple Asphyxiant	Exposure via inhalation of the gas. Signs and symptoms of acute exposure may include rapid respiration, muscular incoordination, fatigue, nausea, vertigo, unconsciousness, and death.
Helium	Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Nitrogen	Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular incoordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Hydrogen	Flammable, Simple Asphyxiate	Oxygen depletion	Exposure via inhalation of the gas. Rapid respiration, muscular in-coordination, fatigue, dizziness, nausea, vomiting, unconsciousness, and death.
Carbon Monoxide	Flammable, poisonous, odorless, Asphyxiate.	Simple Asphyxiant	Exposure via inhalation of the gas. Acts on blood causing damage to central nervous system. Can be fatal even with adequate oxygen. Can form explosive mixtures with air. Symptoms of exposure include shortness of breath, headache, confusion, nausea, dizziness, and unconsciousness.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Agilent 7890A (or similar) GC equipped with a Front TCD and an Aux TCD

6.1.2. Automated data system capable of archiving instrument runs

6.1.2.1. Chemstation is the data acquisition system.

6.1.2.2. Chrom version 2.1 is the data processing system.

6.2. Supplies

6.2.1. Chromatographic grade stainless steel and/or nickel tubing and chromatographic grade stainless steel connective fittings (Valco, SwageLok, or equivalent)

6.2.2. Stainless steel chromatographic packed columns – HaySep N 1.8 m X 1/8” X 2.0 mm 60/80; Mol Sieve 5A 1.8 m X 1/8” X 2.0 mm 45/60

6.2.3. Assortment of gas-tight syringes from 0.01-mL to 2.0-L volume for standard preparation (Hamilton, SGE, or equivalent)

6.2.4. Pressure regulators for carrier gas, Front TCD/Aux TCD, and standards – single-stage, stainless steel diaphragm

6.2.5. Stainless steel vacuum/pressure gauge capable of measuring from -30 inches of mercury to 30 psig, or transducer and process meter capable of measuring from 0 psia to 50 psia

6.2.6. 7-micron filters (Nupro or equivalent)

6.2.7. Air sample bag, 1- or 3-L (SKC or equivalent)

6.2.8. Passivated canister, 1.0-L, 1.8-L, 6-L or 15-L (SIS, Restek, Anderson Instruments, Rasmussen, or equivalent)

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP He for carrier gas, TCD reference gas, sample preparation, and standard preparation

7.1.2. UHP H_2 for detector combustion and the reduction catalyst

7.1.3. Zero-grade air for detector combustion and the oxidation catalyst

7.1.4. UHP N2 for carrier gas, TCD make-up gas, TCD reference gas, sample preparation, and standard preparation

7.2. Primary Gas Standards

7.2.1. Primary gas standards (CH_4, CO_2, CO, N_2, He, H2, and O_2) are available in various concentrations as mixtures or pure components, and analytically certified by the supplier (Scott-Marrin, Scott Specialty, or equivalent).

7.2.2. Dilutions of primary gas standards are made on a volume/volume basis using serial dilution methodologies. UHP N_2 or UHP He is used as the diluent gas.

7.3. Working gas standards

7.3.1. Working gas standards are prepared in passivated canisters or air sample bags by making dilutions of the primary gas standards. See Attachment 3 for the nominal concentration levels used in the initial calibration (ICAL).

7.3.2. Working Gas Standard Prepared in Canisters - To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated passivated canister. The aliquot is metered in by measuring the vacuum of the canister as the aliquot is being transferred. The ratio of the final canister pressure and the pressure transferred is the dilution factor. The canister is pressurized with UHP N2 or UHP He and is then allowed to equilibrate.

7.3.3. Working Gas Standard Prepared in Air Sample Bags - To prepare a working gas standard, an aliquot of the primary gas standard or of the pre-mixed primary gas standard is transferred to a clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N2 or UHP He to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.4. To calculate the concentration of the standard prepared:

$$\text{ppmv} = C_{ps} \times P_i / P_f$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL (if using air sample bag)

P_f = final pressure of canister in psia or final volume in mL (if using air sample bag)

7.4. The preparation of each standard is recorded in the Laboratory Information Management System (LIMS).

7.5. Expiration date for standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to TestAmerica SOP WS-QA-0017 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders. Expiration of working standards is six months.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements:

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
Passivated Canister	2000 mL	None	30 days	Advisory – Active Soil Gas Investigations FINAL, April 30, 2012 (DTSC and LARWQCB)
Air Sample Bag	500 mL	None	72 hours	N/A

8.2. Passivated canisters used for sample collection must be certified clean (see SOP WS-QA-0032). A 7-micron filter should be placed on the inlet of the canister to protect the valve from particulates.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. The pressure of a canister should be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.6. Samples are stored at room temperature.

8.7. **Important Note:** The information in this section is the minimum laboratory requirements and is communicated to the client via the Sample Acceptance Policy delineated in the section of the QAM that discusses Handling of Samples.

9. QUALITY CONTROL

9.1. Batch

A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS) and a Method Blank, but they do not count towards the maximum 20 samples in a batch.

9.1.1. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.1.2. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch count.

9.1.3. Laboratory QC samples, including duplicates and clean canister blanks (screen cans), do not add to the batch count.

9.1.4. In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied.

9.1.5. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

9.1.6. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Laboratory Control Sample

For each batch of samples, an LCS must be analyzed after the calibration standard and before the method blank and samples. The LCS is spiked with all the target analytes at concentrations within the calibration range of the method.

9.2.1. If any analyte is outside established recovery and precision control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the LCS or the batch. See also the troubleshooting guidelines in Section 11.5.

9.2.1.1. Repeated failures are an indication of a <u>systematic deficiency</u> that must be corrected.

9.2.1.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the LCS and/or LCSD after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

9.2.1.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the entire batch must be re-analyzed starting with a new initial calibration (ICAL) or a new continuing calibration verification (CCV), depending on whether maintenance or repair was performed.

9.2.2. The LCS/LCSD that failed acceptance criteria should be further evaluated as follows as per the QC program document (QA-PQA-003).

9.2.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include evaluation of sporadic marginal exceedances (ME), or an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.2.4. Current LCS/LCSD control limits are stored in the LIMS. Control limits are subject to change based on periodic evaluation of LCS/LCSD control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.3. Method Blank

For each batch, an acceptable method blank must be analyzed. The method blank is analyzed after the calibration standards and LCS and prior to client samples. The method blank is typically UHP He collected in either a passivated canister or an air sample bag, or may be obtained directly from the laboratory's He supply line. The method blank for H2 and He analysis is UHP N2. The method blank for CH4 (FID), C2H4, C2H6, and C2H2 analysis is UHP He.

9.3.1. The Method Blank must <u>not</u> contain any analyte of interest ≥ RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements. Otherwise, the Method Blank is further evaluated and corrective actions must be performed,

as stated below. See troubleshooting guidelines in Section 11.5.

9.3.1.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

9.3.1.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the method blank after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to sub-ambient method blank pressure, closed method blank container valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

9.3.1.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the entire batch must be re-analyzed starting with a new ICAL or a new CCV, depending on whether maintenance or repair was performed.

9.3.2. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.3.3. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.3.4. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.3.4.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager (PM) must record the client's decision in the NCM.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to CA-Q-P-003, "Calibration Curves & Selection of Calibration Points".

10.2. Initial Calibration

10.2.1. Instruments must be calibrated at initial setup and as needed thereafter, and at least annually. More frequent calibrations must be performed if project-specific requirements dictate.

10.2.2. Prior to sample analysis, an ICAL curve consisting of a minimum of five points at different concentrations is analyzed to determine the linear working range of the analytical system for each target analyte. The lowest calibration level for each target analyte must be at or below the RL. The highest calibration level is considered to represent the upper range of the analytical system for the corresponding target analyte.

10.2.3. The acceptability of the calibration curve for each target analyte is determined by evaluating the percent relative standard deviation (%RSD) of the average CF or RF calculated from the different calibration levels, or by evaluating the correlation coefficient (r) or the coefficient of determination (r2) of the curve.

10.2.3.1. For calibration by average RF, the calibration is deemed acceptable for a given analyte if the %RSD is ≤ 25 (or ≤ 20 for EPA 3C analysis). Peak area is used for this determination.

10.2.3.2. A calibration curve based on linear regression fit is acceptable if r ≥ 0.995 and a minimum of six points is required.

10.2.3.3. A calibration curve based on quadratic (second-order) fit is acceptable if r2 ≥ 0.990 and a minimum of six points is required.

Note: The data acquisition system used for this GC analysis requires forcing the analytical curve through zero. Otherwise, all detections below the lowest point of the calibration curve will not be quantitated.

10.2.4. If the %RSD, r, or r2 does not meet the required acceptance criteria, the corresponding curve is considered invalid and corrective action must be performed. See troubleshooting guidelines and techniques in Section 11.5.

10.2.4.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

10.2.4.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the specific calibration level(s) after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard container valve, incorrect or broken sample line used, detector turned off, or low GC supply gas

pressure.

10.2.4.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, all calibration levels must be re-analyzed and re-evaluated to determine if the %RSD, r, or r2 meets acceptance criteria.

10.2.5. A new ICAL is required when situations like, but not limited to, the following are encountered:

- After changes are made to the original ICAL instrument configuration
- After replacement of analytical columns
- After replacement of detectors

10.2.6. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.2.7. Initial Calibration Verification (ICV)

Each new multi-point calibration must be verified using a second-source standard. The second-source standard, as quantitated against the new calibration curve, must have recoveries between 80% and 120% for each target analyte. If these criteria are not met, the following corrective actions must be performed:

- Rerun the second-source check standard.
- Re-prepare or acquire a new standard.
- Evaluate instrument conditions or perform maintenance or repair, if needed.
- Re-analyze a new ICAL.

10.2.8. An acceptable ICAL and ICV is documented using a GC Initial Calibration Curve Review Checklist (see Attachment 6). This checklist is submitted to the Department Manager, or designee, for second-level review and signature. Only after successful completion of first and second-level reviews may the analysis be reported from the new ICAL.

10.3. Continuing Calibration Verification

10.3.1. A batch may be started after an approved ICAL and ICV have been completed.

10.3.2. An opening CCV standard is analyzed at the onset of each batch, or every 24-hour shift, whichever comes first, to verify the linearity of the ICAL. The CCV will contain each analyte of interest, on each applicable detector, a different source from that used in the ICV. The system is considered in control if the percent difference (%D) between the RF of the analyte in the CCV and the average RF of the analyte in the ICAL is ± 20 (for both ASTM D1946 and EPA 3C analysis).

10.3.3. If the CCV fails acceptance criteria, corrective actions must be performed. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10e, pages 216 and 217 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92), if routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated. See troubleshooting guidelines in Section 11.5.

10.3.3.1. Repeated failures are an indication of a systematic deficiency that must be corrected.

10.3.3.2. For instances when the analytical system is still in control (i.e., analytical system performance is otherwise nominal and no systematic deficiencies are indicated), corrective action is defined as re-analyzing the CCV after documenting and correcting the exact condition that caused the failure. Such failure may be due, but not limited, to poor standard preparation, low standard pressure, closed standard valve, incorrect or broken sample line used, detector turned off, or low GC supply gas pressure.

10.3.3.3. For instances when the analytical system is not in control (i.e., analytical system performance indicates a systematic deficiency or other defect requiring instrument maintenance or repair), corrective action is defined as the performance of instrument maintenance or repair. After maintenance or repair, the CCV (if this still meets the NELAC or TNI requirement stated in Section 9.2.4.3) may be re-analyzed. Otherwise, a new ICAL is required.

10.3.4. Following the last injected sample, the batch is closed with a passing CCV, which must also meet the same acceptance criteria as the opening CCV. Per the NELAC Standard (Quality Systems, June 5, 2003, Section 5.5.5.10c, page 216 of 324) and TNI Standard (EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2c, page 92), acceptable opening and closing CCVs must bracket reportable samples.

10.3.4.1. If the closing CCV fails acceptance criteria, follow the corrective action procedures defined in Section 10.3.3.

10.3.4.2. If any target analyte or target group in the closing CCV exceeds acceptance criteria and the target analyte or target group was ND in the associated samples, no further corrective action is required. However, the nonconformance must be reported in an NCM.

10.3.4.3. If any sample result is reported from a batch where the closing CCV failed acceptance criteria, the CCV failure must be documented in an NCM. The NCM must indicate the reason why the sample results, in the best judgment of the analyst, are being reported under a failed closing CCV.

10.3.5. If sample analysis must be halted for more than an 8-hour hour period, an opening CCV must be analyzed prior to continuance of the batch, to ensure that instrument conditions have remained stable.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation

11.2.1. For air sample bag samples, the air sample bag is checked for damage and is analyzed as received.

11.2.2. For passivated canister samples, the initial pressure is checked by attaching the process meter line connector to the passivated canister. The process meter line connector must be rinsed before use, with the pressurization gas (UHP N_2 or UHP He, if requested) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the process meter line connector attached, the passivated canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the passivated canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in

the Canister Pressurization Logbook (see Attachment 7) and in the individual Canister Field Data Record (see example form in SOP WS-QA-0032).

11.2.2.1. If there is an associated analytical test (e.g., EPA TO-14A or EPA TO-15) to the sample that requires use of a mass flow controller to measure injected sample volume, the passivated canisters should be pressurized with UHP N2 only.

11.2.2.2. If UHP N2 is used, then the final GC results must be corrected for N2 content (see Section 11.7).

11.2.2.3. If N2 is also a target analyte, a back calculation/correction factor is used (see Section 11.7 and 12.8.9).

11.2.3. When the passivated canister vacuum/pressure is increased, a dilution factor is calculated and is applied to the results. The calculation is provided in Section 12.8.6.

11.2.4. Pressurizing canister sample with UHP He is an option, if requested by clients. Pressurizing samples with UHP He will eliminate the error associated with N2 correction. EPA TO-14A or EPA TO-15 analysis on samples pressurized with UHP He will require a flow correction factor.

11.3. Calibration

11.3.1. Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.3.2. Refer to Section 10 for details regarding instrument calibration.

11.4. Sample Analysis

The calibration standards and the sample QC are analyzed in the same manner as client samples. After the calibration standards are analyzed and evaluated (Section 10), the sample QC is analyzed and evaluated (Section 9), all prior to client sample analysis.

11.4.1. Recommend instrument conditions are presented in Attachment 2. If, after instrument maintenance or repair, these parameters change, then the updated parameters must be listed in the instrument maintenance log. The appropriate QC is performed prior to analysis of samples to verify method performance.

11.4.2. For analysis using EPA 3C, all samples must be analyzed in duplicate. The results are acceptable when the peak areas for two consecutive injections

agree within 5% of their average (<10% difference between the sample runs). Note that only client samples are analyzed in duplicate. All calibration and sample QC are analyzed once.

11.4.2.1. If the duplicate runs exceed the acceptance criteria, run the sample one more time to confirm that the excessive variation is due to the sample matrix/container and not the analytical system. Otherwise, perform corrective action based on the investigated cause of the variation. See similar corrective actions defined in Section 9.

11.5. Troubleshooting Guidelines and Techniques

11.5.1. Low pressure in canisters containing the standards or problems with carrier/detector gas supply: Always confirm that adequate pressure remains in the canisters and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.5.2. Low Response: Causes are typically leaking detector.

11.5.3. Baseline Noise: Check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over.

11.5.4. Valve Switching Issues: Excessive baseline disruptions can be caused by valve actuation when the columns are not properly balanced. Balance the columns by adjusting the carrier restrictors so that head pressure does not change when the column is taken offline.

11.5.5. Instrument Issues: If data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.6. Maintenance or Repair of Analytical Instruments or Support Equipment

11.6.1. When analytical instruments or support equipment require repair or maintenance, they shall be taken out of operation or otherwise isolated, and tagged as 'out-of-service' until such a time as the repairs or maintenance have been made and the instrument or support equipment can be demonstrated as operational by calibration and/or verification or other tests to demonstrate acceptable performance. Details on the tag-out procedures to be followed may be found in the section of the QAM that discusses Equipment and Calibrations.

11.6.2. Schedule for routine maintenance of analytical instruments may be found in

Attachment 4.

11.6.3. All maintenance or repair must be documented in the Instrument Maintenance Logbook (see example in Attachment 8).

11.7. Monochrom

11.7.1. Monochrom is used for samples being analyzed for D3588/EPA 3C. Samples being analyzed by these methods need to be run through Monochrom for specific calculations needed for those methods. Refer to the Monochrom manual located on the intranet for specific instructions on how to use the Monochrom program.

12. CALCULATIONS/DATA REDUCTION

12.1. After the analytical run has ended, a real-time review of the data is performed to determine if a successful analysis has been achieved. Peaks which exceed the upper calibration range and/or which exceed the signal output of the detector must be diluted and re-analyzed.

12.2. The chromatogram/data is reviewed to determine if the proper dilution has been performed. If the sample was analyzed at the proper dilution, the chromatogram/data is evaluated to determine if proper peak identification and integration was performed.

12.3. A target compound is identified by comparison of the sample analyte RT with that of the standards analyzed within the corresponding batch.

12.4. When a compound has been identified, that compound will be quantitated based on its RF from the ICAL, the integrated area of the peak as determined by the instrument data system, the dilution factor, and the N_2 correction (if applicable).

12.4.1. The data system automatically quantitates the sample results. If a canister sample was pressurized before analysis, the results must be multiplied by the DF (see Section 12.8.7). Correction for N2 (if applicable) must also be performed (see Section 12.8.9).

12.4.2. If, in the best judgment of the analyst, the instrument data system integration is incorrect, then the analyst may manually integrate the peak. See Section 12.7.

12.5. The laboratory reviews the total or summation of the fixed gases reported as part of the data validation process. The review is a secondary check to confirm that valid data acquisition occurred during analysis. The assessment of 95-100% v/v criteria can provide useful information when the analysis is for the full list of fixed gases, but is not required when a subset of compounds is analyzed or reported. If the total fixed gases

result is below this range, it is possible that the difference is due to the amount of non-analyzed components in the sample. This case must be investigated. Screening data, NMOC data, subsequent H_2 and He analysis, sulfur compounds analysis, and/or other information should be considered. If the sample containers were pressurized with UHP N_2, then they should be evaluated for error associated with UHP N_2 pressurization. Samples that do not meet the required total fixed gases acceptance criteria and which deviation is not yet accounted for, must be re-analyzed at least once.

12.5.1. An investigation of the deviation from the total or summation acceptance criteria may result into transducer recalibration, instrument maintenance, and/or instrument recalibration. Samples must be re-analyzed after the condition has been remedied. The area supervisor, or designee, should be notified as soon as possible when re-analysis is expected to be outside the sample holding time.

12.5.2. Every time the acceptance criteria for the total or summation of fixed gases result are not met, the deviation and the resulting investigation must be reported in an NCM. The same reporting procedure must be followed for unexplained deviation.

12.6. Second-column confirmation for the results is not required due to the unique separation properties of the multiple column analytical system and the limited possibilities of non-analyte interference.

12.7. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.8. Calculations

12.8.1. Calculation for RF

$$\text{RF} = \frac{\text{Peak Area in Standard}}{\text{Concentration of Standard}}$$

12.8.2. Calculation for RPD

$$\text{RPD} = \frac{|\text{Value A - Value B}|}{\text{Average of Values}} \times 100$$

12.8.3. Calculation for %RSD

$$\%\text{RSD} = \frac{\text{Std. Dev of RFs}}{\text{Mean of RFs}} \times 100$$

12.8.4. Calculation for %D

$$\%D = \frac{RF\ ICAL - RF\ CCV}{RF\ ICAL} X 100$$

12.8.5. Calculation for %Rec

$$\%\ \mathrm{Re}\,c = \frac{Amount\ cpd.\ recovered}{Amount\ cpd.\ spiked} X\ 100$$

12.8.6. Calculation for Pressure DF

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = absolute canister pressure before dilution (initial pressure)

Y_a = absolute canister pressure after dilution (final pressure)

12.8.7. Calculation for determining concentration of compound in sample, using average RF

$$\mathrm{Conc.\,Compound} = \frac{\mathrm{Area\ Compound}}{\mathrm{Ave\ RF\ for\ Compound}} \times \mathrm{DF}$$

12.8.8. Reporting units are typically % v/v for ASTM D-1946 and EPA 3C analysis. If results are to be reported in units of ug/L (also mg/m³), use the following equation:

$$\mathrm{Result\ (ppmv)} \times \frac{\mathrm{Molecular\ weight\ of\ compound}}{24.45} = \mathrm{ug/L}$$

Note: 24.45 is the volume of ideal gas at 25º C and 1 atmosphere

1% v/v = 10000 ppmv

12.8.9. Calculation for N_2 results in samples pressurized with N_2:

Total amount of N_2:

$$T_{N2}\ (\text{as analyzed}) = S_{N2}(I/F) + 100((F-I)/F)$$

Where:

T_{N2} is the result % v/v of N_2 as analyzed (no DF applied)

S_{N2} is the N_2 in the sample (no DF applied)

I and F are the initial pressure and the final pressure, respectively, of the sample during pressurization (psia)

100 is the percent concentration of N_2 added

Rearranging,

$$S_{N2} = (T_{N2} - 100((F-I)/F))F/I$$

Or, in final form with the DF applied:

N_2 reported, % v/v = N_2 total (as analyzed, with DF) – [100 x (DF-1)]

12.9. No conversion of the analytical results to standard conditions is made.

12.10. Calculations for D1945 (Natural Gas)

This calculation is performed automatically by Monochrom.

Before performing the calculations for D1945, the following parameters must be determined:

D1946	TO3	EPA 15_16
CO2, O2, N2, CH4, CO, H2, He	Ethane, Propane, Butane, Pentane, C5 Range, C6 Range, C7 Range, C8 Range, C9 Range, C10+ Range	H2S (optional)

12.10.1. Results for each of the above parameters are normalized by first summing the raw % v/v results for each parameter to determine the total %. The values are then normalized:

$$\text{Normalized Result} = \frac{\text{Raw Result} \times 100}{\text{Total \%}}$$

When the normalized results are summed, they should equal 100%.

Non-detect values are presumed to be 0 for the purposes of this calculation.

12.11. Calculations for D3588

This calculation is performed automatically by Monochrom.

Before performing the calculations for D3588, the following parameters must be determined, and the normalization as described above performed.

D1946	TO3	EPA 15_16
CO2, O2, N2, CH4, CO, H2, He	Ethane, Propane, Butane, Pentane, C5 Range, C6 Range, C7 Range, C8 Range, C9 Range, C10+ Range	H2S (optional)

12.11.1. Three properties are determined for D3588 – Ideal Gross Heating Value, Ideal Net Heating Value, and Specific Gravity. For each property, the contribution

from the measured values is calculated by using the property constant and the normalized result:

$$\text{Contribution} = \frac{\text{Normalized Result} * \text{Property Constant}}{100}$$

Units for Gross Heating Value and Net Heating Value are btu/cubic ft.

Parameter	Gross Heating Value Constant	Net Heating Value Constant	Specific Gravity
Carbon Dioxide	0	0	1.5196
Oxygen	0	0	1.1048
Nitrogen	0	0	0.96723
Methane	1010	909.4	0.55392
Carbon Monoxide	320.5	320.5	0.968
Ethane	1769.7	1618.7	1.0382
Propane	2516.1	2314.9	1.5226
n-Butane	3262.3	3010.8	2.0068
n-Pentane	4008.9	3703.9	2.4912
C5 Balance	4008.9	3703.9	2.4912
C6 CPDS	4755.9	4403.9	2.9755
C7 CPDS	5502.5	5100.3	3.4598
C8 CPDS	6248.9	5796.2	3.9441
C9 CPDS	6996.5	6493.6	4.4284
>/= C10 CPDS	7742.9	7189.9	4.9127
Hydrogen Sulfide	637.1	586.8	1.1767
HYDROGEN	324.2	273.93	0.6960
Helium	0	0	0.13820

12.11.2. Once individual values are calculated, the results for each property are summed and reported.

12.12. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of appropriate review checklists are found in Attachments 5 and 6.

12.12.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for air, soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the CCV/LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste

disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.1.1. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood. Once empty, they are turned over to the Hazardous Waste Specialist and damaged (e.g. a hole drilled into the cylinders so they cannot be reused, then they are disposed or recycled.

15.1.2. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.2. Air sample bags are slashed in a hood and allowed to completely vent, then placed into an orange high VOA lab trash can. When the can is full or after no longer than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum for the incinerator in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment..

16. REFERENCES/CROSS REFERENCES

16.1. ASTM D 1946-90 (Re-approved 2000), Standard Practice for the Analysis of Reformed Gas by Gas Chromatography.

16.2. EPA 3C, Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from stationary sources, 40 CFR Chapter 1, Part 60, Appendix A.

16.3. TestAmerica Sacramento QAM, current revision

16.4. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.5. Advisory – Active Soil Gas Investigations, January 28, 2003 (DTSC and LARWQCB)

16.6. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.7. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.8. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.9. TestAmerica Corporate SOP CA-Q-S-002, Manual Integrations, current revision

16.10. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

16.11. TestAmerica Sacramento Safety SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision.

16.12. ASTM D 1945-03 (Re-approved 2010), Standard Test Method for Analysis of Natural Gas by Gas Chromatography

16.13. ASTM D 3588-98 (Re-approved 2011), Standard Practice for Calculating Heat Value, Compressibility Factor, and Relative Density of Gaseous Fuels.

17. METHOD MODIFICATIONS

17.1. ASTM D-1946

17.1.1. The method uses a calibration method in which a separate standard is run closely approximating each sample's concentration for target analytes. TestAmerica Sacramento uses the more widely accepted method of initial multi-point and continuing calibrations to define linear response.

17.1.2. TestAmerica Sacramento does not normalize sample results to 100%, unless required by the client. Normalizing assumes a zero contribution of all other components not analyzed (e.g., H2, He, hydrogen sulfide, etc.), and can affect results significantly.

17.1.3. Commercially-prepared standards that have the industry standard analytical uncertainty of 2-5% are used.

17.1.4. Standards may be prepared in N_2 as well as in He.

17.1.5. The method specifies use of a TCD.

17.1.6. Due to the multi-detector, multi-valve configuration of TestAmerica Sacramento's analytical instrument for fixed gases analysis, one or more sample loops, not necessarily equal to 0.5 mL, may be used.

17.2. EPA 3C

17.2.1. The method specifies use of at least three concentration levels for the multi-point calibration, spanning the range of suspected sample concentrations. TestAmerica Sacramento uses a minimum of five-point calibration on each applicable detector for all standard analyte components, to define the reporting range.

17.2.2. TestAmerica Sacramento does not use correction for moisture content. This would require additional client measurements to be supplied, and at most would result in a 2-3% correction in reported values.

17.2.3. TestAmerica Sacramento does not use correction for temperature at sampling. As the laboratory supplies fully evacuated sample containers for sampling, any field temperature does not affect results. Laboratory temperature is constant for sample pressurization and analysis.

17.2.4. The method specifies that "helium must be used to prepare calibration gases" so that detector response is consistent. TestAmerica Sacramento also uses UHP N2, UHP helium, or zero-grade air to prepare calibration gases. Detector response is unaffected by the diluent gas since the analytical system separates the diluent gas from the standard components prior to detection. Furthermore, sample injection size is determined by a sample loop and not a mass flow controller.

17.2.5. The method specifies that if two consecutive sample injections do not agree within 5% of their average, then run additional samples until consistent area data are obtained. TestAmerica Sacramento requires that only at least one additional injection be analyzed. Refer to Section 11.4.5.

18. ATTACHMENTS

18.1. Attachment 1: Target Analytes and Reporting Limits

18.2. Attachment 2: GC Conditions – Agilent 7890A

18.3. Attachment 3: Nominal ICAL Concentrations

18.4. Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

18.5. Attachment 5: Example GC Initial Calibration Curve Review Checklist

18.6. Attachment 6: Example GC Technical Data Review Checklist

18.7. Attachment 7: Canister Pressurization Logbook (Example Page)

19. REVISION HISTORY

19.1. WS-GCA-0020, Revision 1.3, Effective 06/09/2016

19.1.1. Changed Section 11.7.1 Monochrom only used for Method 3C analytes.

19.1.2. Editorial changes.

19.2. WS-GCA-0020, Revision 1.2, Effective 07/25/2014

19.2.1. Added references to D3588 and D1945 in the title and Section 16.

19.2.2. Sections 12.10 and 12.11 were inserted to support the calculations for D3588 and D1945.

19.2.3. Editorial Changes.

19.3. WS-GCA-0020 Revision 1.1, Effective 05/28/2014

19.3.1. Changed Section 2.1 to include – "Carbon monoxide (CO) is last to elute from the HaySep, bypassing the isolated Mol Sieve, then goes to the TCD. Ultra high purity (UHP) helium (He) is used as the carrier gas.

19.3.2. Updated Table in Section 5.2 to include Carbon Monoxide.

19.3.3. Editorial changes.

19.4. WS-GCA-0020, Revision 1, Effective 6/19/2013

19.4.1. Included reference to Chrom as the data processing software in Section 6.1

19.4.2. Change references to SOP LA-QAS-002 and LA-SRA-002 to refer to the appropriate Sacramento SOPs (WS-QA-0017 and WS-QA-0032, respectively).

19.4.3. The first sentence of Section 12.5 has been revised from "The laboratory acceptance criteria for the total or summation of the fixed gases reported must be equal to or greater than 95% v/v, not to exceed 105% v/v." to "The laboratory reviews the total or summation of the fixed gases reported as part of the data validation process. The review is a secondary check to confirm that valid data acquisition occurred during analysis. The assessment of 95-100% v/v criteria can provide useful information when the analysis is for the full list of fixed gases, but is not required when a subset of compounds is analyzed or reported."

19.4.4. Inserted "required" into the last sentence of Section 12.5.

19.4.5. Replaced Attachments 5, 6, and 7 with Sacramento-specific examples.

19.4.6. Removed Attachment 8 (example maintenance logbook).

19.4.7. Updated Attachment 3 with current calibration concentrations.

19.4.8. Inserted section 9.1.4, "In some cases, an LCS Duplicate may be required by a

client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied."

19.5. WS-GCA-0020, Revision 0, Effective 04/01/2013

19.5.1. This is the first version of this SOP.

Attachment 1: Target Analytes and Reporting Limits

Compound	RL, % v/v[1]
Carbon Dioxide	0.50
Carbon Monoxide	0.010
Methane	0.50
Nitrogen	1.0
Oxygen	0.20
Helium	0.10
Hydrogen	0.010

Attachment 2: GC Conditions – Agilent 7890A

Fixed Column Oven: 100°C
FID: Oven 250°C, FID Range = 12, Time Constant = Slow
TCD: Oven 130°C, Filament 210°C, Range = 0.5, Polarity = positive, Time constant = slow
Reduction Catalyst: 380°C
Sample Lines and A/S valve at 80°C
Valve Ovens at 120°C
Fixed FF = 20 mL/min, BF = 30 mL/min w/ He reference at 20 mL/min
H_2/He FF=20 mL/min, BF = 20 mL/min w/ N_2 reference at 20 mL/min
FID H_2 20 mL/min + 10 mL/min Red Cat H_2
FID Air = 300 mL/min
Initial time t = 0, all events off
0.01 min: Sample flow starts
1.7 min Sample flow stops with 0.2 min before loop vent
2 min Sample loop injection
3.05 min Mol Sieve Column Isolation
3.5 min H_2/He stripper BF for 4 min relay event time
6.5 min Fixed BF
6.7 min Fixed Mol Sieve in line
Additional events include catalyst recharge and sample advance pre-purge

[1] Note that RLs are subject to change based on annual MDL studies.

Attachment 3: Recommended Nominal ICAL Concentrations (%v/v)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Carbon Dioxide	0.01	0.02	0.05	0.2	1.0	5	10	50	100
Carbon Monoxide	0.01	0.02	0.05	0.2	1.0	5	10	50	100
Methane - TCD	0.50	1.0	5.0	10	50	-	-	-	-
Nitrogen	1.0	10	30	80	100	-	-	-	-
Oxygen	0.2	1.0	10	20	30		-	-	-
Helium	0.02	0.1	0.2	1.0	10	50	-	-	-
Hydrogen	0.010	0.1	0.5	1.0	10	50	-	-	-

Attachment 4: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.
Quarterly	Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed.
As needed	All Detectors: Clean when baseline indicates contamination or when response is low. Perform periodic leak checks (quarterly). Replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds). Bake trap to correct for high background. Replace gas supply. Clean moisture filters. Bake column.

Attachment 5: Example GC Initial Calibration Curve Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

GCA ICAL Review Checklist

Instrument ID: _____ Method: _____

Work List ID: _____ Analysis Batch(es): _____

Analytes Included in this ICAL: _____

ICAL Review	1st Level	2nd Level	N/A
1. Method locked in Chrom.			
2. ICAL is set as most recent method in Chrom.			
3. RTO Marker included for and RT TO3 windows set correctly.			
4. Standards used in ICAL are current.			
5. No analytes have saturated peaks.			
6. All peaks correctly identified and within retention time windows.			
7. Manual integrations reviewed and appropriate.			
8. All analytes meet %RSD or linear regression requirements.			
9. Any levels dropped are reviewed and appropriate.			
10. ICV meets requirements and is run after the ICAL.			
11. RFs are calculated correctly. Perform Manual calculation.			
12. ICAL meets requirements stated in SOP.			
13. Chromatograms are uploaded/reviewed in TALs.			
14. ICAL is locked in TALs.			

Curve Valid for:	Yes	No	Criteria
Standard SOP			RSD ≤20%, r≥ 0.995 (intercept <1/2RL for 8081,8082, NQ) and (<RL for 8330) and r>0.990 (intercept <RL for 8015), ICV≤15%, PEM ≤15%
DOD QSM V3,V4, V5			RSD ≤20%, r≥ 0.995, ICV≤20%, PEM ≤15%

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Comments: _____

_____

_____

_____

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Attachment 6: Example GC Technical Data Review Checklist

TestAmerica

Sacramento

Air GC Data Review Checklist

Job Number(s): _____ Instrument/Work List ID(s): _____

TALS Analytical Batch(es): _____ ICAL Batch(es): _____

☐ TO3 ☐ TO12 ☐ 1946/3C ☐ EPA 15/16

Review Items	— Level 1 — Yes	No	N/A	Level 2
Initial Calibration				
1. Is ICAL locked in Chrom and in TALS?				
2. If RRF used for ICAL, did it meet acceptance criteria? ☐ 20% (8000 series) ☐ ≥ 0.990 (EPA 1516) ☐ 30% (TO-12)				
3. Is ICV properly linked in TALS for Tier 4 jobs ?				
Continuing Calibration				
1. Did %Drift (%D) meet method criteria (30% TO-12) (15% EPA 15/166) (20% QSM-TO-3/1946)?				
2. Are compounds within retention time windows?				
3. Does CCV frequency meet criteria (10 samples QSM) or every 20 samples (standard work)?				
4. RTC present for TO3 analytes?				
Client Samples & QC Sample Results				
1. Was the correct analysis performed & were project instructions followed?				
2. Were preparation and analysis completed within holding times?				
3. Are preparation weights/volumes entered correctly?				
4. Have Chromatograms been uploaded/reviewed?				
5. Are positive results within calibration range?				
6. Are any dilutions due to matrix? (Requires NCM if "yes.")				
7. Are all positive results within RT windows, both columns if 2 required?				
8. Are target constituents in LCS/LCSD within control limits?				
9. All target analytes in MB < RL (< ½ RL for DoD)? (Requires NCM if "no.")				
10. Do results (e.g. dilutions) make sense?				
11. Are all manual integrations appropriate and documented properly?				
12. Are nonconformances documented as NCMs?				
13. Are all QC samples properly linked in TALS?				
14. Have all flags been reviewed for appropriateness?				
15. Are all Chrom graphics and reports reviewed/uploaded in the analytical batch?				
16. Has QC checker been run?				
17. NCM numbers:				

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Comments: _____

_____

_____

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Attachment 7: Canister Pressurization Logbook (Example Page)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

Canister Pressurization Log

Date	Sample ID	Can #	Initial Press. (psig)	Initial Press. (psia)	Final Press. (psig)	Final Press. (psia)	Gauge ID	Initial	Comments
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		

A = Gauge CD5437; B = Gauge _____ [Calculation: psig + 14.7 = psia]

QA-813 r1 GEC 7/21/2014 PAGE 1 Reviewed by /Date: _____

Title: Determination of Low-Level Volatile Organics in Ambient / Indoor Whole Air Samples Using GC/MS-Scan Mode

[Methods EPA TO-14A and EPA TO-15]

Approvals (Signature/Date):

Koroush Vaziri Department Manager	7-22-15 Date	Joe Schairer Health & Safety Manager / Coordinator	7/22/15 Date
Lisa Stafford Quality Assurance Manager	7/24/2015 Date	Crystal Pollock Laboratory Director	7.24.15

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the analysis of low-level volatile organic compounds (VOCs), having molecular weight in the general range of 40-200 g/mol and vapor pressure greater than 0.10 Torr at 25°C and 760 mm Hg in ambient air, by gas chromatography/mass spectroscopy (GC/MS) technique. This SOP is based on the EPA TO-14A/TO-15 method specifications and is applicable to various air matrices that include ambient air and indoor air.

1.2. Target analytes and reporting limits with this SOP are listed in Attachment 1. Reporting limits will be proportionately higher for samples that require dilution.

1.3. On occasion, clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the laboratory's Quality Assurance Manual (WS-QAM) in the section that discusses Service to the Client

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. An air sample and internal standards (IS) are metered by a mass flow controller onto a cryogenically cooled trap (using either a Microscale Purge and Trap or a Cold Trap Dehydration technique described in Section 11). The trap is heated and the contents are transferred to a Tenax trap to remove water. The Tenax trap is heated and the analytes are transferred to a cryofocusing module. The cryofocuser is heated to transfer the analytes to the gas chromatographic column for separation and detection by a mass spectrometer operated in the Scan Mode.

3. DEFINITIONS

3.1. Note that "must" and "shall" in this SOP denote required activities.

3.2. Air Sample Bag: Commonly referred to as FlexFilm or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (e.g., SKC or ESS).

Note: Use of air sample bags as sample collection media constitutes a modification to the method (see Section 17.5) that is defined in the final report.

3.3. Part per billion volume to volume (ppbv or ppb v/v): Concentration expressed as part of gaseous (vapor) volume of pure target compound contained in a billion part of gaseous volume of sample.

Note: This reporting unit is NOT equivalent to the common ppb unit used in soil or water analysis.

3.4. Particulate Filter: A cylindrical stainless steel fitting containing a fritted metal disc, which is connected to the valve of a passivated canister or to the vacuum flow regulator (VFR), to prevent particulate matter from entering and damaging the passivated canister or VFR.

3.5. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-L, 1.8-L, 6-L, 15-L, or 33-L volumes.

3.5.1. SUMMA canister: A spherical stainless steel container, of which the interior has been specially treated by a process (SUMMA passivation), that renders all surfaces inert to VOCs.

3.5.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.5.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of VOCs during storage.

3.6. Standard molar volume = 24.45 L/mol at standard conditions (i.e., room temperature of 25°C and standard pressure of 1 atmosphere).

3.7. Standard pressure = 1.0 atmosphere or 14.6 pounds per square inch absolute (psia) or 0 inches of mercury or 0 pound per square inch gauge (psig), based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = -30 inches of mercury vacuum

3.8. QC section: Surrogates: Organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Although not required by the method, each client and QC sample is spiked with surrogate standards via the analytical trap. Surrogates are used to monitor method performance with each sample. Surrogates are only reported to the client by request.

3.9. Vacuum Flow Regulator: A device which, when connected to a passivated canister, regulates the flow of sample into the passivated canister so that a timed, representative sample can be obtained (also called a composite sample), as opposed to an unregulated, instantaneous sample (grab sample).

3.10. Vacuum/Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from -30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia (psig + 14.7 = psia).

3.11. Further definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, a target analyte in this SOP. Before using Ultra High Purity Nitrogen (UHP N2), hydrocarbon-free air, IS mix, or target compound standard mix cylinders, each regulator should be purged with the appropriate gas.

4.2. Contamination may occur in the sampling system if passivated canisters are not properly cleaned prior to use. Passivated canisters shall not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above the RL, or a level previously agreed upon between the laboratory and the client. When more stringent canister cleaning acceptance criteria are warranted based on project-specific or regulatory requirements, and then the more stringent criteria must be used. Further information regarding the cleaning and certification of passivated canisters may be found in TestAmerica SOP WS-QA-0032. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2.1. Passivated canisters may be batch-certified or individually-certified, depending on client request.

4.2.2. Passivated canisters will be certified-clean down to the MDL of the target analytes of interest if sample results need to be evaluated down to those limits. However, the laboratory must be provided advanced notification of the requirement.

4.2.2.1. Common laboratory contaminants like Acetone and Methylene chloride may be present above the MDL. In this instance, client approval must be received prior to sending out these passivated canisters.

4.3. Carry-over may occur when samples with high levels of contaminants are analyzed. The sample immediately following a high-level sample shall be re-analyzed if carry-over is suspected.

4.4. Air sample bags may contain low levels of target analytes.

4.5. Only compounds having both similar mass spectrum and GC retention time (RT) would be expected to interfere in the method. This situation most commonly occurs with structural isomers.

4.6. Large concentrations of water, Methane, or Carbon dioxide may limit the size of the sample aliquot that can be effectively cryo-trapped. This may elevate the RLs for samples of this type.

4.7. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interference will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Pressurized gas equipment is used in this procedure. Be sure all valves and gauges are operating properly and that no equipment is over-pressurized. After changing cylinders, check all gas line connectors for leaks, with soapy water. Release of high pressure gas can cause rapid suffocation.

5.1.2. This analysis may require transfer of the sample from an air sample bag to a canister (Section 11.2). Because of the flexible nature of the air sample bag, insertion of the syringe needle into the bag may cause the septa to flex or the shaft of the syringe needle may flex within the septa. This may allow sample to escape along the shaft of the needle. All samples being manually removed from an air sample bag and transferred into a canister must be handled inside a fume hood with chemical protective gloves, lab coat, and safety glasses.

5.1.3. When pressurizing canisters or changing cylinders, face shield must be worn over safety glasses.

5.1.3.1. Passivated canisters should never be pressurized over 40 psig.

5.1.4. Pressurized gas cylinders must be securely retained. The use of a face shield is required when changing regulators.

5.1.5. Air sample bags should not be pressurized, as seam splitting will result.

5.1.6. The preparation of standards and reagents will be performed in a fume hood with the sash set at the level indicated on the side of the hood.

5.1.7. Both the GC and the MS contain elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the instrument.

5.1.7.1. Temperature-appropriate gloves must be worn when working with hot or cold items.

5.1.8. Latex and vinyl gloves provide no protection against organic solvents. Nitrile or similar gloves must be used.

5.1.9. The effluents from the sample splitters for the GC and the roughing pumps for the MS must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.1.10. The MS is under deep vacuum and must be brought to atmospheric pressure before working on the source.

5.1.11. Due to high voltage risk, power to the GC and/or MS must be turned off or disconnected before work can be done on the instrument.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Helium	Simple Asphyxiant	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses.
Liquid Nitrogen	Simple Asphyxiant Cryogenic liquid	NA – Keep oxygen levels at 19.5%	Oxygen Deficient atmosphere may cause headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting and depression of all the senses. Contact with skin may cause frostbite-changes in skin color to white or grayish-yellow.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Instrumentation

6.1.1. Gas chromatograph – capable of sub-ambient temperature programming and electronic pressure control (Hewlett Packard 6890 or equivalent).

6.1.2. Mass-selective detector – equipped with computer and appropriate software (Hewlett Packard 5973/5975 or equivalent with Chemstation data acquisition software).

6.1.3. Sample concentrator – equipped with a cryogenic trap and appropriate systems for the control of moisture (Entech 7100 or equivalent).

6.1.4. Chrom version 2.1 data processing software.

6.2. Supplies

6.2.1. Chromatographic grade stainless steel or nickel tubing and stainless steel plumbing fittings.

6.2.2. Chromatographic column – Rtx-Volatiles, 0.32 mm ID, 1.5 µm df, 60 m length, methyl polysilicate liquid phase (Restek Corporation or equivalent).

6.2.3. Transducer and process meter capable of measuring 0 psia to 50 psia, for preparing standards (Ashcroft Digital Vacuum/Pressure Gauge or equivalent).

6.2.3.1. The process meter must be calibrated quarterly, at a minimum, against the master gauge.

6.2.4. Pressure regulators for carrier gas and standards – 2-stage, stainless steel diaphragm (single stage acceptable for standards).

6.2.5. Stainless steel vacuum/pressure gauge capable of measuring from -30 inches of mercury to 40 psig (Span Instruments or equivalent).

6.2.6. Air sample bags and passivated canisters used for the preparation of standards and the dilution of samples.

6.2.7. Screen can for preparation of method blanks. This is a cleaned canister certified to be free of analytes at levels greater than or equal to the MDL or levels specified by client or program requirements, whichever is appropriate to the samples being analyzed.

6.2.8. Gas-tight syringes of various sizes (Hamilton or equivalent).

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP N_2 – used for Method Blanks and preparing dilutions of samples and standards

7.1.2. UHP Helium – used as the gas chromatograph carrier gas

7.1.3. Pressurized air source for Entech 7100 heater gas

7.1.4. Liquid N_2

7.1.5. Distilled or NanoPure water

7.2. Standards

7.2.1. Gas calibration stock standards containing the target compounds, at a nominal concentration of 1 part per million volume/volume (ppmv or ppm v/v), are purchased from NIST-approved vendors or prepared from neat in passivated canisters. Suppliers are required to provide certification of the analyte concentrations.

7.2.2. IS and surrogate stock standard mix (see Attachments 5 and 6, respectively), at a concentration of 300 ppbv, are purchased from NIST-approved vendors. Suppliers are required to provide certification of the analyte concentrations.

7.2.2.1. The surrogate mix is also used to tune the mass spectrometer.

7.3. Standard Preparation

Static dilutions and other standard preparation activities are performed in accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Static dilutions of the stock standard gas mixtures are made in 6- or 15-L passivated canisters to create working standards. A high precision vacuum gauge is flushed with UHP N_2 and attached to the top valve of a clean, evacuated passivated canister, and the absolute pressure is recorded.

7.3.1.1. Distilled or NanoPure water (50 µL) is added to calibration standards prior to mixing.

7.3.1.2. The IS mix does not contain water.

7.3.2. Depending on the concentration of each stock standard gas mixture, a particular pressure of each is added to the passivated canister to achieve the

desired concentration in the working standard.

7.3.2.1. As an example, the daily working standard, at a nominal concentration of 10 ppbv, is created by adding 5.0 psia of the 100 ppbv standard mix and adding UHP N_2 to the passivated canister to achieve a final pressure of 50 psia.

7.3.2.2. Care should be taken to flush each regulator and transfer line with standard prior to transfer to the passivated canister.

7.3.3. Detailed preparation of each standard is recorded in the Laboratory Information Management System (LIMS) reagent module.

7.3.4. Other preparation techniques may be used to obtain the desired standard concentration, provided these techniques do not compromise the integrity of the standards used, and that the details of the preparation are properly documented in the LIMS.

7.3.5. Working standards are valid for a period of 30 days, after which fresh standards are prepared.

7.4. Expiration dates for source standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to TestAmerica SOP WS-QA-0017 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample container, preservation techniques, and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Passivated Canister	2000 mL	None	30 days	Advisory – Active Soil Gas Investigations, April, 2012 (DTSC, LARWQCB, and SFRWQCB)
Air Sample Bag	500 mL	None	72 hours	N/A

8.2. Passivated canisters used for sample collection must be certified clean (< RL or program specific limit). Canisters are cleaned in accordance with TestAmerica SOP WS-QA-0032. Filters (e.g., 7-micron or 2-micron) should be placed on the inlet of the canister to protect the valve from particulates.

8.3. Samples should be shipped at room temperature, in packaging suitable to prevent puncture and exposure to light.

8.4. If air sample bags are to be shipped by aircraft, they should be filled about 75% full to allow for expansion during shipment.

8.5. The pressure of a passivated canister should be recorded before and after sample collection in the field to help detect canister leakage and document proper sampling.

8.6. Samples are stored at room temperature.

8.7. Samples should be protected from extreme temperatures.

9. QUALITY CONTROL

9.1. Batch

A batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS) and a Method Blank, but they do not count towards the maximum 20 samples in a batch.

9.1.1. In some cases, an LCS Duplicate may be required by a client or program to provide batch precision. In that instance, the acceptance criteria and corrective actions appropriate for the LCS are applied.

9.1.2. Rerun of the same client sample is counted as part of the 20 in a batch (i.e., a client sample analyzed twice in the same batch must be counted as two client samples).

9.1.3. Field quality control (QC) samples (e.g., trip blanks, equipment blanks, and field duplicates) count as client samples; therefore, they add to the batch

count.

9.1.4. Laboratory QC samples, including duplicates and clean canister blanks (screen cans), do not add to the batch count.

9.1.5. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

9.1.6. Refer to the laboratory's QC Program document (WS-PQA-003) for further details of the batch definition.

9.2. Laboratory Control Sample– For each batch, an LCS must be analyzed. The LCS is analyzed after the calibration standards and before the Method Blank and client samples. The LCS is spiked with the target analytes in Attachment 1, from which a sub-set may be reported..

9.2.1. Refer to the QC Program document (WS-PQA-003) for details on the requirements for LCS composition.

9.2.2. If any analyte is outside established recovery and precision control limits, or any surrogate is outside the established recovery control limits, the system is out of control and corrective action must occur. Corrective action typically includes reanalysis of the batch.

9.2.3. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable reasons for not reanalyzing include evaluation of sporadic marginal exceedances (ME), or an elevated recovery (indicating a high bias) with samples non-detect for the failing analyte. Refer to the QC program document (WS-PQA-003) for more details regarding evaluation and acceptance of out of control LCS data.

9.2.4. Exceedance outside the ME limits require corrective action, regardless of whether the associated result is positive or ND. See Section 9.2.6.

9.2.4.1. For failures that exceeded the ME at the high end, the ND analyte may be flagged and reported only if the program or project-specific requirements allow.

9.2.4.2. For all other ND results that failed the ME requirements, the client must approve to flag and report the data since the nonconformance does not meet the NELAC or TNI Standard.

9.2.5. All data reported with out of control LCS values will be flagged by the LIMS. Analysts shall also file a nonconformance memo (NCM) within the LIMS

detailing why the data is reported, and any corrective actions performed.

9.2.6. Corrective actions to occur before batch reanalysis:

9.2.6.1. Evaluate the analytical run for errors and anomalies. Re-analyze the LCS.

9.2.6.2. Consult the troubleshooting guidelines in Section 11.8. Evaluate the instrument status and perform maintenance.

9.2.6.3. Re-analyze the continuing calibration verification (CCV) standard and LCS, or recalibrate.

9.2.7. Current LCS control limits are stored in the LIMS. Control and ME limits are subject to change based on periodic evaluation of LCS control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.3. Method Blank

9.3.1. For each batch, an acceptable Method Blank must be analyzed. The Method Blank is analyzed after the calibration standards and LCS and prior to client samples. The Method Blank is a 6-L screen can (Section 6.2.7) humidified with 50 µL of Distilled or NanoPure water and then pressurized to 40 psia with UHP N_2.

9.3.2. If a method blank is requested to be analyzed using an air sample bag similar to which samples are collected the client is required to submit a bag for that purpose. The laboratory does not maintain an inventory of air sample bags, therefore, Method blanks in bags is by client request only.

9.3.3. The Method Blank must <u>not</u> contain any analyte of interest ≥ RL (or ≥1/2 RL, as dictated by the QSM or project-specific requirements), except common laboratory contaminants, (Section 9.3.2). Otherwise, the Method Blank is further evaluated and corrective actions must be performed, as stated below. See troubleshooting guidelines in Section 11.8.

9.3.3.1. Re-analyze the Method Blank once to determine if an error or an anomaly occurred during sample analysis. If the re-analysis is acceptable, then the Method Blank can be considered in control.

9.3.3.2. If there are no results greater than the RL in the samples or if the results in the samples are greater than 10X the Method Blank level, the data may be reported with qualifiers. In this case, the elevated Method Blank result is not believed to impact data quality. The anomaly must be reported in an NCM.

9.3.3.3. If there are results greater than the RL in the samples and if these results are less than 10X the Method Blank level, the samples must be re-analyzed.

9.3.3.3.1. If re-analysis is not possible due to limited sample volume or other constraints, the Method Blank is reported and all associated samples are flagged. The client must be consulted. The anomaly must be reported in an NCM. The laboratory Project Manager (PM) must record the client's decision in the NCM.

9.3.4. If the analyte detected in the Method Blank is a common laboratory contaminant (Methylene chloride, Acetone, and 2-Butanone), the data may be reported with qualifiers if the concentration of the analyte is less than 5X times the RL. Otherwise, corrective actions, as stated in Section 9.3.1, must be performed. The anomaly must be reported in an NCM.

9.3.5. If surrogates are a project-specific requirement, then the Method Blank must have acceptable surrogate recoveries. If surrogate recoveries are unacceptable, the data must be evaluated to determine if the Method Blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-analysis of the Method Blank and affected samples must be performed.

9.4. Surrogate Standards.

Surrogates are not a method requirement. The laboratory routinely adds surrogates to all QC and client samples via the analytical trap and will report these results only if defined in a project/contract or at client's request. The surrogate compounds used in this SOP are listed in Attachment 6.

Surrogate recoveries in QC and client samples may be assessed to ensure that recoveries are within laboratory control limits. If any surrogate is outside these limits and if surrogates are a project-specific requirement, the following corrective actions must be performed:

- Check all calculations for error.
- Ensure that instrument performance is acceptable. See troubleshooting guidelines in Section 11.8.
- Re-analyze the QC/client sample.

9.4.1. It is only necessary to re-analyze a client sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst has reason to believe that the repeated out of control results are due to problems other than matrix effect.

9.4.2. If re-analysis is not possible due to limited sample volume or other constraints, the surrogates are reported with a flag. The client must be consulted. The anomaly must be reported in an NCM. The nature of the matrix interference must be noted in the NCM. The PM must record the client's decision in the NCM.

9.4.3. Current surrogate control limits are stored in the LIMS and are subject to change based on periodic evaluation of surrogate control charts by Quality Assurance personnel, in accordance with the procedures detailed in policy WS-PQA-003.

9.5. Internal standard (IS)

IS compounds are added to each calibration standard, LCS, Method Blank, and client sample via the analytical trap. IS compounds are monitored for each shift by comparing the IS areas and retention times in each client and QC sample against those of the associated CCV standard. The IS compounds used in this SOP are listed in Attachment 5.

9.5.1. IS evaluation criteria for the initial calibration (ICAL) may be found in Section 10.3.8.

9.5.2. For all other QC and client samples, IS areas are considered acceptable if they fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A Low-level) of the CCV IS areas. The RTs are considered acceptable if they fall within ±20 seconds (±0.33 minutes) of the IS RT of the associated CCV.

9.5.3. Any QC or client sample exceeding the acceptance criteria above must be re-analyzed. If the IS fails upon re-analysis, the failure must be documented in an NCM. All corrective actions performed must also be documented in the NCM.

9.6. Sample Duplicate Analysis

9.6.1. A client sample duplicate is analyzed and reported with the batch when requested by the client.

9.6.2. The acceptance criteria for the duplicate analysis is an RPD ≤ 25 for target analytes detected >5X the RL. No criteria are established for duplicate results <5X the RL.

9.7. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed,

and return to control in the Instrument Maintenance Logbook. Section 11.8 lists troubleshooting guidelines.

9.7.1. Refer to WS-PQA-021 for corrective actions specific to DoD programs.

10. CALIBRATION

For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.1. Initial/Daily Tuning of the Instrument

10.1.1. After a successful autotune per manufacturer's recommendations, each instrument is manually tuned using perfluorotributylamine (PFTBA) so that mass-to-charge ratio (m/z) 69 is 100%, m/z 131 is approximately 34%, and m/z 219 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day.

10.1.2. At the beginning of each 24-hour shift, prior to any analytical runs, it must be verified that the GC/MS system meets acceptable tune performance criteria. This is done through the analysis of 50 ng of 4-bromofluorobenzene (BFB); the acceptance criteria are listed in Attachment 3 (for TO-14A) or in Attachment 4 (for TO-15).

10.1.2.1. Using the BFB method in the cryotrap software, the IS mixture volume required is 50 ng of BFB on column (currently 24 mL of 300 ppbv standard).

10.1.2.2. An alternate way to load 50ng BFB is to use 50mL of a 20ppbv IS/SURR mix and 20mL of a 3000ppbv IS/SURR mix.

10.1.2.3. The mass spectrum of BFB must be acquired using the peak apex and the scans immediately before and after the apex and averaged. A background subtraction is applied using a scan prior to the elution of BFB.

10.1.3. If any of the key ions fail the ion abundance criteria listed in the attachments, the system is considered out of tune and any subsequent sample/standard analysis shall be considered unacceptable. The BFB must be re-analyzed and re-evaluated. If the BFB continues to fail, the GC/MS system must be evaluated. See troubleshooting guidelines in Section 11.8.

10.1.3.1. Adjustments to the mass axis calibration, the electron multiplier voltage, or other tune parameters may be required. All parameter changes must be recorded in the Instrument Maintenance Logbook.

10.1.4. BFB tunes may be analyzed more frequently depending on documented client requirements.

10.2. Initial Calibration

10.2.1. Instruments are calibrated at initial setup and as needed thereafter, and at least annually.

10.2.2. An ICAL curve consisting of a minimum of five points is analyzed to determine the linear working range of the analytical system for each compound. An average response factor (RF), or sometimes called the relative response factor (RRF), and the percent relative standard deviation (%RSD) are calculated for each target analyte using the equations in Section 12.5.

10.2.3. The ICAL is considered acceptable if the calculated %RSD for the RF (or RRF) for each analyte not listed in Section 10.3.4 is <30, with at most two exceptions up to a limit of 40%.

10.2.4. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %RSD limit up to 55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.2.5. Linear calibration using least squares regression may be used with the appropriate number of calibration points. Details regarding its use and the calculations involved may be found in TestAmerica Corporate QA SOP CA-Q-S-005. This Corporate SOP must be consulted prior to using this curve. The analyst must read and understand the topics regarding Forcing Through Zero and Curve Weighting.

10.2.5.1. The coefficient of determination for a line fit must be greater than or equal to 0.990

10.2.5.2. The absolute value of the intercept (printed on the calibration curve plot in Chrom) should be less than ½ the reporting limit (-RL ≤ intercept ≤ +RL). If the intercept is outside the limits, any values

below the reporting limit must be evaluated to ensure that false positives or false negatives are not being reported.

10.2.6. If the ICAL acceptance criteria are not met, corrective action (documented in the Instrument Maintenance Logbook) must be performed and a new ICAL generated. See troubleshooting guidelines in Section 11.8.

10.2.7. The nominal concentrations of the ICAL standards are typically 0.20, 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, and 40 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.20, 0.40, and 0.80 ppbv standards are analyzed by trapping 62.5, 125, and 250 mL, respectively, of a 0.80 ppbv working standard.

10.2.7.1. At times, the default volume may be changed, depending on required instrument sensitivity for a project.

10.2.8. Internal Standards in the ICAL

10.2.8.1. The IS response at each calibration level must fall between 60% and 140% (for TO-15) or –50% and 200% (for TO-14A) of the IS response in the mid-point calibration standard. The mid-point standard is normally 8ppbv.

10.2.8.2. The RT shift for each of the IS at each calibration level must be within ±20 seconds (0.33 minutes) of the RT of the IS in the mid-point calibration standard.

10.2.8.3. Any calibration level exceeding the above acceptance criteria must be re-analyzed.

10.2.9. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation, in accordance with Policy CA-T-P-002, Selection of Calibration Points.

10.3. Essential components for ICAL evaluation

10.3.1. The signal-to-noise (S/N) in the low point of the ICAL must be ≥ 2.5:1 for the compound to be considered valid at that level. Evaluate the EICP for the S/N determination.

10.3.2. Qualitative compound identification criteria must be met for all calibration levels; (primary and secondary ions >10% must be present in each standard level. Consult the technical director or QA for clarification.

10.3.3. Check retention times for isomers to ensure correct peak assignment.

10.3.4. Check co-eluting peaks for the proper reference spectrum.

10.3.5. The 're-fit' or 'read-back' for each point of the calibration curve is evaluated for the % error. The guideline is ≤ 20% for each level of the curve. This can be observed in the calibration summary in Chrom for each compound.

10.4. Initial Calibration Verification (ICV)

10.4.1. Each new ICAL must be verified using a second-source standard.

10.4.2. Since the regulatory agencies have not provided guidance on second-source verification, the ICV is considered acceptable if the %Recovery for each analyte not listed in Section 10.4.3 is 70–130%, with at most two exceptions up to a limit of 60–140%.

10.4.3. Any four of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %Recovery of 45–155:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.4.4. For samples analyzed in the same batch where the ICAL and ICV were analyzed, every time an allowed exception to the 70–130% Recovery ICV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.4.5. If the ICV acceptance criteria are not met, the following corrective actions must be performed. See troubleshooting guidelines in Section 11.8.

10.4.5.1. Rerun the second-source check standard.

10.4.5.2. Re-prepare or acquire a new standard.

10.4.5.3. Evaluate instrument conditions.

10.4.5.4. Regenerate a new ICAL.

10.4.6. Due to the limited availability of second-source manufacturers for the air standard mixes and some neat compounds, the following options may be considered as second-source:

10.4.6.1. Different certified lot from the same manufacturer.

10.4.6.2. Same certified lot from the same manufacturer but the stock standard used for the second-source is prepared by an analyst other than the one who prepared the stock standard used for the ICAL. This option is only allowed if the program or project-specific requirements allow.

10.5. Continuing Calibration Verification

10.5.1. Unless the QC batch follows a new ICAL and an ICV, for every 24 hours of operation, a CCV standard is analyzed to verify the ICAL average RF. The %D of the CCV RF from the ICAL average RF is calculated for each target analyte using the equation in Section 12.5.4.

10.5.2. The CCV is considered acceptable if the %D for each analyte not listed in Section 10.5.3 is ±30.

10.5.3. Any <u>four</u> of the following analytes, which are not listed as target analytes in the published methods and are considered poor performers, may have a %D ±55:

1,1,2-Trichloro-1,2,2-trifluoroethane	Dichlorodifluoromethane
1,2,3-Trichlorobenzene	Naphthalene
2-Hexanone	Propene
Acetone	Tetrahydrofuran
alpha-Methylstyrene	

10.5.4. Any time an allowed exception to the ±30%D CCV criteria is used, the sample results (whether J-value, ND, or positive) for the affected analyte must be flagged and explained in an NCM.

10.5.5. The following NELAC requirements (NELAC Quality Systems, June 5, 2003, 5.5.5.10e, page 217 of 324) and TNI Standard EL-V1M4-2009, Quality Systems for Chemical Testing, Section 1.7.2e, page 92) apply when the CCV acceptance criteria are not met.

10.5.5.1. If routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated.

10.5.5.2. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was ND in the associated samples, the ND analyte may be reported with a flag. An NCM must be generated and the high bias discussed in the case narrative of the final report.

10.5.5.3. When the acceptance criteria for an analyte in the CCV are exceeded high (i.e., high bias), and the analyte was detected at a positive hit in the sample, the sample must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted.

10.5.5.4. When the acceptance criteria for an analyte in the CCV are exceeded low (i.e., low bias), sample results may be reported if they exceed a maximum regulatory limit/decision level (if known). Otherwise, the affected samples must be re-analyzed after a passing CCV or after a new ICAL has been established, evaluated, and accepted. An NCM must be generated and the low bias discussed in the case narrative of the final report.

10.5.6. CCVs may be analyzed more frequently depending on documented client requirements.

10.5.7. CCVs must be monitored on a routine basis using the control chart program (refer to SOP WS-QA-0035) to evaluate the data for trends. The frequency is dependent on the frequency of the analysis. In the LIMS control chart module, select the CCV chart. Once the chart has been evaluated, click “Save Log” to save the chart evaluation in the LIMS.

10.6. Calibration standards and other QC samples (e.g., BFB, LCS, Method Blank, etc.) may not be analyzed more than twice without documented corrective action. If the initial run fails acceptance criteria, re-inject the calibration standard or QC sample. If second run passes, analysis may proceed. Otherwise, conduct instrument maintenance or perform corrective action. Completely document failure, corrective action performed, and return to control in the Instrument Maintenance Logbook. Section 11.9 lists troubleshooting guidelines.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Preparation – Air Bag Samples

11.2.1. For air sample bag samples, the air sample bag is checked for damage and is analyzed as received.

11.2.2. Air sample bags are analyzed directly from the bag or transferred to an evacuated can within 72 hours of sampling.

11.2.2.1. If the entire bag is transferred to a can, the bag needle valve septum is pierced with a needle attached to a 1-L or a 6-L evacuated can, and the entire contents transferred.

11.2.2.2. If only a portion of the bag is to be transferred, a measured aliquot of the bag is transferred via a clean syringe through a septum attached to the top of a 1-L or a 6-L humidified can.

11.2.3. After transfer, the can is then pressurized to a positive pressure and the pressure is recorded.

11.3. Sample Preparation – Canister Samples

11.3.1. For passivated canister samples, the initial pressure is checked by attaching the process meter line connector to the passivated canister. The process meter line connector must be rinsed before use, with the pressurization gas (UHP N_2 or UHP He, if requested) by physically holding it against the gas outlet and flushing for 10 seconds, as this avoids possible carry-over concerns from high concentration samples. With the process meter line connector attached, the passivated canister valve is opened briefly and the pressure is recorded. If the pressure is less than 6 psig, the passivated canister is pressurized to 10 psig with the pressurization gas. The initial and final pressure must be recorded in the Canister Pressurization Logbook (see Attachment 10) and in the individual Canister Field Data Record.

11.3.1.1. Samples received above ambient pressure (14.6 psia) do not require pressurization unless additional volume is needed to perform multiple analyses. If samples are received below ambient pressure, UHP N_2 should be added. The default final pressure is 24 to 26 psia, however, the final pressure should be above ambient but not more than three times the initial pressure.

11.3.2. When the passivated canister vacuum/pressure is increased, a dilution factor (DF) is calculated and is applied to results. The calculation is provided in Section 12.5.5.

11.3.3. Passivated canisters received as trip blanks (without sample collected) are pressurized to 24-26 psia. These samples are considered to have a DF =1.0.

11.4. Sample Screening

Samples are screened to check for contamination before analysis or if suspected to contain significant contamination, using a GC/MS. Screening is performed to determine a proper dilution or the optimum volume of sample for the calibrated range, and to prevent overloading the analytical instrument. The screening instrument is generally calibrated at a single-point for common analytes of interest. The sample screen data are stored in TALS within the Chrom module.

11.5. Tuning

Refer to section 10.2 for details regarding instrument tuning.

11.6. Calibration

11.6.1. Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

11.6.2. Refer to Sections 10.3 through 10.5 for details regarding instrument calibration.

11.7. Sample Analysis

11.7.1. The calibration standards and the sample QC are analyzed in the same manner as client samples. After the calibration standards are analyzed and evaluated (Sections 10.3 through 10.5), the LCS is analyzed and evaluated (Section 9.2), and then the Method Blank is analyzed and evaluated (see Section 9.3), all prior to client sample analysis.

11.7.2. Each passivated canister is attached to the autosampler and recorded in the instrument sequence. A sequence is created in the GC/MS software to prepare the instrument for data acquisition. The sequence information controls the GC/MS method, data file creation, sample parameters, and report output. A second sequence must be created in the autosampler control software to control the sampling process such as line position, sample volume, trap temperatures, flow rates, and times. The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified.

11.7.3. The valves are opened on all passivated canisters and the autosampler and GC/MS sequences are started.

11.7.4. The pressure DF must be compensated for by trapping more than the default volume. For example, a sample received at 12.0 psia and pressurized to 24.6 psia has a pressure DF of 2.05. If the default volume is 250 mL, then 510 mL should be trapped. The recorded volume is rounded to three significant figures.

11.7.5. A sample that requires only a small dilution can be analyzed by trapping a volume less than the standard volume. The minimum volumes that can be trapped are 10 mL for an Entech 7200 and 20 mL for an Entech 7100. The maximum volume that can be trapped is three times the default volume. Larger dilutions may require analysis using different methodology.

11.7.6. Sample dilutions may also be performed by transferring an aliquot of the sample (originally from either an air sample bag or a passivated canister) into an air sample bag and filling it up to volume or by removing an appropriate amount of the original pressurized sample from a passivated canister and then re-pressurizing to approximately 24 to 26 psia or no more than 3x the initial pressure with UHP N_2. Serial dilutions can be performed, as necessary.

11.7.6.1. Air sample bag dilutions can be used for reporting analytes that exceeded calibration range in the original analysis, but not as a reportable analysis for all other analytes due to the low-level contamination inherent in the air sample bag.

11.7.6.2. When air sample bag dilutions are performed, the syringe used for transferring sample must be fitted with a valve (e.g., Luer-Lok) that allows the syringe contents to be isolated from the room air during transfer between containers.

11.7.7. For routine analysis, either the Microscale Purge and Trap or the Cold Trap Dehydration technique is used.

11.7.7.1. For Microscale Purge and Trap technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.1.1. Glass Bead trap (Module 1) is cooled to -150°C

11.7.7.1.2. Internal standard is trapped

11.7.7.1.3. Sample is trapped

11.7.7.1.4. Tenax trap (Module 2) is cooled to -15°C and the Glass Bead trap is heated to 10°C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on

instrument performance. This step is designed to remove water from the sample.

11.7.7.1.5. When GC is ready, the cryofocuser (Module 3) is cooled to –170°C. Module 2 is heated to 200°C. The sample is transferred to Module 3.

11.7.7.1.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.1.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.7.2. For Cold Trap Dehydration technique, the autosampler will follow the sequence of events below (parameters may be modified based on instrument performance):

11.7.7.2.1. Blank (empty) trap (Module 1) is cooled to -40°C and the Tenax trap (Module 2) is cooled to -40°C.

11.7.7.2.2. Internal standard is trapped.

11.7.7.2.3. Sample is trapped.

11.7.7.2.4. Blank trap is heated to 10°C. Any remaining sample is transferred to Module 2 by passing UHP Helium through Module 1. Conditions may vary based on instrument performance. This step is designed to remove water from the sample.

11.7.7.2.5. When GC is ready, the cryofocuser (Module 3) is cooled to –155°C. Module 2 is heated to 200°C. The sample is transferred to Module 3.

11.7.7.2.6. Module 3 is heated and the GC/MS column flow is routed through Module 3 to inject the sample and begin the run.

11.7.7.2.7. The system is pre-flushed with the next sample and the system is baked to limit carry-over.

11.7.8. Upon completion of the analytical sequence, the Entech software generates a QA/QC report that records data from the sampling event (i.e. actual volume trapped, temperature at the time of trapping, sample pressure, etc.).

11.8. Troubleshooting Guidelines

11.8.1. Many problems encountered during analysis are due to low standard pressures or carrier/detector gas supply issues. Always confirm that adequate pressure remains in the standards and that the instrument gas supplies are sufficient before working on the instrument hardware.

11.8.2. Low response – typically caused by leaking sample lines or valves or contaminated/dirty sources. Instrument software can perform automated leak checks of the system. Specific components can be checked by isolating the component in question from the system (disconnect and cap or plug the ends) and then performing a leak test using a pressure gauge and passivated canister at positive pressure. Leaking components will not hold pressure when the passivated canister is closed. Low internal standard areas may be caused by degradation of the MS performance and increasing the electron multiplier (EM) voltage may solve this concern.

11.8.3. Baseline noise – check for supply gas contamination and leaking fittings. Carrier gas filters may need to be changed, including the pencil filters inside the GC. Sample carry-over or contamination may also be an issue and baking the system while flushing sample lines will remove most carry-over. A dirty source or leaking MS may also cause issues. The use of automated leak check routines in the MS software can indicate if a leak is present. Source-cleaning should be performed according to the manufacturer's instructions.

11.8.4. Tune issues – if an instrument will not pass tune the first step is to perform a mass axis calibration and peak-width adjustment. If the failure is due to ratios of ions with large differences, the tune parameters should be adjusted to achieve the desired ratios. The final corrective action is to clean the source according to the manufacturer's instructions.

11.8.5. Instrument issues – if data loss or error messages are encountered, consult the instrument troubleshooting guidance found in the operator's manual. The manual is in the help section of the GC software.

11.9. Maintenance or Repair of Analytical Instruments or Support Equipment

11.9.1. When analytical instruments or support equipment require repair or maintenance, they shall be taken out of operation or otherwise isolated, and tagged as 'out-of-service' until such a time as the repairs or maintenance have been made and the instrument or support equipment can be demonstrated as operational by calibration and/or verification or other tests to demonstrate acceptable performance. Details on the tag-out procedures to be followed may be found in the section of the QAM that discusses Equipment and Calibrations.

11.9.2. A new ICAL must be generated following major maintenance such as

changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing the concentrator.

11.9.3. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments (instrument contains two filaments under vacuum), replacing the syringe or injector tower, changing/refilling the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps or transfer lines.

11.9.4. Schedule for routine maintenance of analytical instruments may be found in Attachment 9.

11.9.5. All maintenance or repair must be documented in the Instrument Maintenance Logbook.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analyses

Two criteria must be satisfied to verify positive identification:

12.1.1. Elution of sample component at the same GC relative or absolute RT as those of the standard component.

12.1.1.1. The sample component relative retention time (RRT) must compare within ±0.06 RRT units of the RRT of the standard component.

12.1.1.2. As an option, RT must compare within 0.33 minutes of the standard component absolute RT. For reference, the RT standard must be run within the same 24-hour shift as the sample.

12.1.2. Correspondence of the sample component and the standard component mass spectra.

12.1.2.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.2.2. The relative intensities of ions must agree within ±30% between the standard reference and sample spectra. For example, for ions with ratio of 50% in the reference spectra, the corresponding sample ratio must be between 20 and 80%.

12.1.2.3. Standard reference mass spectra must be obtained on each individual GC/MS system.

12.1.3. If an analyte cannot be verified by all of the criteria in the above sections, but in the technical judgment of the analyst the identification is correct, then the analyte may be reported

12.1.3.1. Technical judgment may be based on whether a compound is present when co-elution occurs and a determination is made based on retention time and mass spectrum.

12.1.3.2. The difference in the spectra and the reason for the decision to report results must be explained in an NCM.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. The following sections identify the guidelines for making tentative identification:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. Relative intensities of the major ions should agree within ±30%.

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.1.6. Only peaks having a total ion current greater than 10% of the nearest eluting IS total ion current will be evaluated for reporting.

12.2.2. TICs will be given general names consisting of major functional groups and number of carbon atoms unless an RT reference is available.

12.2.3. When TICs are requested to be reported using specific compound names, the following procedure must be followed:

12.2.3.1. Choose characterized ions of the specific compounds from the mass spectrum.

12.2.3.2. Search ions from expected RT range or entire RT range if the RT of the specific compound is unknown or uncertain.

12.2.4. Semi-quantitative results will be calculated for TICs using total ion current areas and assuming an RRF = 1.0.

12.2.5. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the analyst assign a tentative identification.

12.3. Quantitative Analysis

12.3.1. When an analyte has been identified, the quantification of that analyte will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Quantitation will take place using the IS technique.

12.3.2. A sample must be analyzed and reported at a dilution if one or more target analytes have an on-column amount above the upper calibration level. Dilutions are acceptable if at least one of the following criteria are met:

12.3.2.1. Any target analyte in the diluted sample is at or above the mid-point calibration standard (e.g., 8 ppbv on-column if 8 ppbv is the mid-point).

12.3.2.2. The peak height of any non-target analyte in the diluted sample exceeds the largest peak height of the highest calibration standard.

12.3.2.3. A heavy hydrocarbon matrix in the diluted sample raises the baseline two times that of the relative IS.

12.3.3. Analyte quantitation must be performed from the ICAL response and not from the CCV response. Test results must be qualified in reports when analyte quantitation is based on the CCV at client's request. This request must also be documented in an NCM and reported in the case narrative of the final report.

12.4. All manual or re-integration of chromatograms must be documented in accordance with TestAmerica Corporate SOP CA-Q-S-002. Documentation includes, at a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a secondary-level review.

12.5. Calculations

12.5.1. Calculation for RPD

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ Values} X\ 100$$

12.5.2. Calculation for RRF

$$RRF = \frac{Area\ cpd\ in\ Std.}{Area\ I.S.} X \frac{Conc.\ I.S.}{Conc.\ cpd\ in\ Std.}$$

The area of the primary quantitation ion is used in the calculation.

I.S. = Internal Standard

12.5.3. Calculation for %RSD

$$\%RSD = \frac{Std.\ Dev.\ of\ RRFs}{Mean\ of\ RRFs} X 100$$

12.5.4. Calculation for %D

$$\%D = \frac{Average\ RRF\ from\ ICAL - RRF\ CCV.}{Average\ RRF\ from\ ICAL} X 100$$

12.5.5. Calculation for pressure DF

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = absolute canister pressure before dilution (initial pressure)

Y_a = absolute canister pressure after dilution (final pressure)

12.5.6. Calculation for Determining the Concentration of Compounds

The data system automatically quantitates the sample results based on a standard sample size of 250 mL. The default result units are in ppbv. If a sample size other than 250 mL was used or a canister sample was pressurized, the result must be adjusted as shown below:

$$Final\ result\ ppbv = raw\ result\ ppbv\ X \frac{250\ mL}{sample\ vol\ injected} X \frac{final\ psia}{initial\ psia}$$

Where:

$$raw result\ ppbv = \frac{Area\ cpd\ in\ sample}{Area\ I.S.\ in\ sample} X \frac{Conc. I.S.}{RRF\ ICAL.}$$

Note: The area of the primary quantitation ion is used in the calculation.
I.S. = Internal Standard

12.5.7. Calculation for Percent Recovery (%Rec)

$$\%\,\mathrm{Re}\,c = \frac{Amount\ cpd.\ recovered}{Amount\ cpd.\ spiked}\ X\ 100$$

12.5.8. Standard reporting units are ppbv (also ppb v/v). If results are to be reported in ng/L or ug/m^3, use the following equation:

$$result\ ppbv\,X\,\frac{Molecular\ weight\ \ of\ cpd}{24.45} = results\ ng/L\ \ or\ ug/m^3$$

Note: 24.45 is the molar volume of ideal gas in liters at 25°C and 1 atmosphere.

12.6. Estimates of uncertainty are based upon LCS historical control limits, and are provided on request only.

12.7. "J" values (results below the RL but above the MDL) are reported on request only.

12.8. No conversion of the analytical results to standard conditions is made.

12.9. The number of significant figures to be used when reporting sample and QC results are defined in QA Policy WS-PQA-004.

12.10. Technical Data Review

Technical data review is performed in accordance with Policy WS-PQA-012, and is documented utilizing review checklists. Examples of appropriate review checklists are found in Attachments 7 and 8.

12.10.1. One aspect of technical review is to ensure that the test instructions are clear, and that all project-specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must consult the Department Manager or the appropriate PM, who must clarify the instructions.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix

B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. This requires analysis of QC check samples containing all of the standard analytes for the method. It may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Expired standards will be part of the Lab Pack waste stream. They will be identified as expired, stored under the manufacturer's recommended conditions, and then packed for disposal as outlined in SOP WS-EHS-0001.

15.1.1. Gas standards in non-returnable, non-refillable cylinders, such as Scotty® Transportables, are slowly vented in the fume hood when empty. They are then turned over to the hazardous materials specialist, who ensures that they are damaged (e.g., a hole is drilled into the cylinders) so they cannot be reused. The damaged cylinders are then either recycled or scrapped.

15.1.2. Gas standards in returnable, refillable cylinders are returned to the manufacturer.

15.2. Air sample bags are slashed in a hood and then placed into an orange high VOA lab trash can. When the can is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd edition, January 1999

16.1.1. Compendium Method TO-14A, Determination of Volatile Organic Compounds (VOCs) in Ambient Air using Specially Prepared Canisters With Subsequent Analysis Gas Chromatographic Analysis

16.1.2. Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

16.2. TestAmerica Sacramento QAM, current revision

16.3. TestAmerica Sacramento SOP WS-QA-0032, Cleaning, Certification, and Preparation of Sampling Equipment, current revision

16.4. TestAmerica Corporate Environmental Health and Safety Manual CW-E-M-001, current revision

16.5. Advisory – Active Soil Gas Investigations, April 2012 (CAEPA, DTSC, LARWQCB, and SFRWQCB)

16.6. EPA/600/R-04/003, 2003 NELAC Standard, June 5, 2003

16.7. The NELAC Institute (TNI) Standard 2009, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis

16.8. TestAmerica Sacramento SOP WS-PQA-001, Quality Control Program, current revision

16.9. TestAmerica Corporate SOP CA-Q-S-005, Calibration Curves (General), current revision

16.10. TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices, current revision

16.11. TestAmerica Sacramento Policy WS-PQA-004, Rounding and Significant Figures, current revision

16.12. TestAmerica Corporate SOP CA-Q-S-006, Detection Limits, current revision

16.13. TestAmerica Sacramento SOP WS-EHS-0001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision

17. METHOD MODIFICATIONS

17.1. Method TO-15 describes the use of zero air when making standards. TestAmerica Sacramento uses UHP nitrogen in the standards preparation.

17.2. UHP Nitrogen is used for dilution/pressurization purposes.

17.3. Method TO-15 describes canisters leak check being performed using pressure. TestAmerica performs the canisters leak check using vacuum unless specified by the project.

17.4. Method TO-15 presents criteria for evaluating canister cleanliness as less than 0.2 ppbv of any target VOC. TestAmerica Sacramento works with clients to ensure that the canisters meet their data quality objectives, by providing canisters evaluated to the reporting limit or method detection limit as required by their program.

17.5. Method TO-14A describes an inlet system that uses a vacuum to pull the sample through the trap. TestAmerica Sacramento optionally uses the pressure of the sample canister to drive the sample through the trap.

17.6. Method TO-14A describes the use of a Nafion dryer to remove excess moisture from air matrices. TestAmerica Sacramento does not use a Nafion dryer since polar compounds may be lost during this removal step.

17.7. Method TO-14A describes the BFB tune check to be a gas sample introduced via a sample loop. TestAmerica Sacramento traps and analyzes BFB using the same analytical technique used with samples.

17.8. Methods TO-14A and TO-15 describe the use of passivated steel canisters for sampling and analysis. No mention is made of the use of air sample bags. TestAmerica Sacramento analyzes samples in air sample bags for VOCs using the same procedures described herein. A modification to the method is noted in the NCM submitted with the final report.

17.9. Methods TO-14A and TO-15 indicate that in order for the ICAL to be acceptable, all compounds must have a %RSD <30 (with allowance for two that could be up to 40% in TO-15). For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the ICAL if up to four of these analytes have a %RSD ≤ 55. See Section 10.3.4. This modification accounts for analytical issues that arise from poor performing analytes.

17.10. For the continuing calibration criteria, Method TO-14A states that the RPD of each RF (in the CCV) from the mean RF of the ICAL curve should be <30%; for Method TO-15, the %D for each target compound must compare to the ICAL at ±30%. For routine analysis, for those analytes not listed in the published methods and are considered poor performers, TestAmerica Sacramento accepts the CCV if up to four of these analytes have a %D ±55. See Section 10.6.3. This modification accounts for analytical issues that arise for poor performing analytes.

17.11. Method TO-15 recommends maintaining control charts of the %D values for CCV standards. Due to limitations of the LIMS control chart module, these control charts are maintained using the % recovery values for the CCVs. This modification still permits monitoring for adverse trends.

17.12. Surrogates are not required by the methods. This SOP adds surrogates to every QC and client sample to help monitor for matrix effects and method performance. These compounds are included in the initial calibration at a single concentration for each calibration point. However, surrogates are not reported unless requested.

17.13. Method TO-15 states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument. See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.

17.14. The transfer of sample from Tedlar bags to canisters is supported by the conclusions of an EPA poster titled LOSS/GAIN OF VOCS FROM TEDLAR BAGS AND OTHER SAMPLING EQUIPMENT, by C. Loss Paul as presented at the Presented at The 17th Annual Association for Environmental Health and Sciences Meeting, San Diego, CA, March 21 - 22, 2007. According to the synopsis,

> Soil gas samples are collected to evaluate human health risk from vapor intrusion into homes and other buildings. In order to meet risk assessment goals, the analytical reporting limit for many compounds of concern are down to part per billion ranges. The appropriate sampling tubing, sample containers and leak check compounds should be selected in order to ensure data quality objectives. Mechanisms which can impact sample integrity include adsorption of volatile organic compounds (VOCs) onto the sampling media and diffusion of VOCs through the sampling media which may result in artificially low values, and desorption of compounds from the sampling media into the sample which may results in artificially high values. A literature

search was conducted to compile published results of impacts on soil gas samples from sampling equipment (i.e., tubing) and containers (i.e., Tedlar® bags). The literature search revealed that the recommended holding time for samples stored in Tedlar® bags not exceed 48 hours. A laboratory study was conducted to evaluate the holding times for certain VOCs in 1 L Tedlar® bags stored at two different temperatures. VOCs used in this experiment include 1,1,1-trichloroethane (1,1,1-TCA), trichloroethylene (TCE), benzene, and toluene that were combined in a gas mixture with nitrogen. Bags were filled with the gas mixture and then stored for different times and temperatures. Two incubators were set at two temperatures, C to simulate field temperatures and the bags were stored from 8°C and 25°C 15 hours to 2 weeks. Results of this study show that 1,1,1-TCA, TCE, and benzene can be stored up to one week without significant impact on concentrations. Results for toluene are less conclusive. However, soil gas samples collected in Tedlar® bags should be analyzed as quickly as practical or samples can be transferred to another container with longer holding times (i.e., Summa canister).

18. ATTACHMENTS

18.1. Attachment 1: Standard Analytes, Reporting Limits, and Characteristic Ions

18.2. Attachment 2: BFB GC Operating Conditions, EPA TO-15

18.3. Attachment 3: BFB Acceptance Criteria, EPA TO-14A

18.4. Attachment 4: BFB Acceptance Criteria, EPA TO-15

18.5. Attachment 5: Internal Standards

18.6. Attachment 6: Surrogate Standards

18.7. Attachment 7: Example GC/MS Initial Calibration Curve Review Checklist

18.8. Attachment 8: Example GC/MS Technical Data Review Checklist

18.9. Attachment 9: Schedule for Routine Maintenance of Analytical Instrument

18.10. Attachment 10: Canister Pressurization Logbook (Example Page)

18.11. Attachment 11: GRO Analysis

19. REVISION HISTORY

19.1. WS-MSA-0015, Revision 1.6, Effective 07/31/2015

19.1.1. Changed Copyright Information statement on Title page.

19.1.2. Table A3 –Changed gasoline calibration standard 1 from 200 ppb v/v to 100

ppb v/v.

19.1.3. Editorial changes.

19.2. WS-MSA-0015, Revision 1.5, Effective 04/03/2015

19.2.1. Changed Section 10.1.2.1 from "28mL of 250pbv" to "24mL of 300ppbv".

19.2.2. Changed Section 10.1.2.2 from: An alternative way to load 50ng BFB is to use 100ml of a 10ppbv IS/SURR mix and 24mL of a 250 IS/SURR" to "An alternate way to load 50ng BFB is to use 50mL of a 20ppbv IS/SURR mix and 20mL of a 3000ppbv IS/SURR mix."

19.2.3. Editorial changes

19.3. WS-MSA-0015, Revision 1.4 Effective 03/06/2015

19.3.1. Added Section 7.3.5 to read, "Working standards are valid for a period of 30 days, after which fresh standards are prepared."

19.3.2. Added Section 10.5.7 to read, "CCVs must be monitored on a routine basis using the control chart program (refer to SOP WS-QA-0035) to evaluate the data for trends. The frequency is dependent on the frequency of the analysis. In the LIMS control chart module, select the CCV chart. Once the chart has been evaluated, click "Save Log" to save the chart evaluation in the LIMS."

19.3.3. Inserted Section 17.4 to read, "Method TO-15 presents criteria for evaluating canister cleanliness as less than 0.2 ppbv of any target VOC. TestAmerica Sacramento works with clients to ensure that the canisters meet their data quality objectives, by providing canisters evaluated to the reporting limit or method detection limit as required by their program."

19.3.4. Inserted Section 17.11 to read, "Method TO-15 recommends maintaining control charts of the %D values for CCV standards. Due to limitations of the LIMS control chart module, these control charts are maintained using the % recovery values for the CCVs. This modification still permits monitoring for adverse trends."

19.3.5. Section 17.1.2, inserted the sentence, "These compounds are included in the initial calibration at a single concentration for each calibration point."

19.4. WS-MSA-0015, Revision 1.3, Effective 08/15/2014

19.4.1. Section 8, revised second row of table to reflect the more recent "Advisory, Active Soil Gas Investigations" (from 2012 rather than 2003). Reference in 16.5 also changed.

19.4.2. Inserted Section 5.1.2, safety requirements when transferring sample from air bags into syringes or canisters.

19.4.3. Section 8 – in accordance with the most recent DTSC advisory, changed the holding time to 30 days for canisters under DTCS requirements as well.

19.4.4. Changed Section 7.2.2 from 'concentration of 250 ppbv…' to 'concentration of 300 ppbv…'.

19.4.5. Changed Section 7.3.1.1 from 125 µL to 50 µL Distilled or Nanopure water.

19.4.6. Inserted Section 9.7.1, referring to DoD requirements.

19.4.7. Added Section 10.2.5.1, "The coefficient of determination for a line fit must be greater than or equal to 0.990

19.4.8. Added Section 10.2.5.2, The absolute value of the intercept (printed on the calibration curve plot in Chrom) should be less than ½ the reporting limit (-RL ≤ intercept ≤ +RL). If the intercept is outside the limits, any values below the reporting limit must be evaluated to ensure that false positives or false negatives are not being reported.

19.4.9. Changed Section 10.2.7 to – "The nominal concentrations of the ICAL standards are typically 0.30, 0.40, 0.80, 2.0, 4.0, 8.0, 20, 40 and 60 ppbv, but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are analyzed by preparing stock standards at the required concentration or by varying the trapped volume of the working standards from the default volume of 250 mL. For example, the 0.30, 0.40, and 0.80 ppbv standards are analyzed by trapping 30, 40, and 80 mL, respectively, of a 2.5 ppbv working standard."

19.4.10. Removed Section 11.2.4, 11.2.4.1, and 11.2.4.2, which discussed the laboratory default to analyze air sample bags at a 20x dilution.

19.4.11. Removed the subheadings from Section 11.4 (Screening).

19.4.12. Section 11.7.2, appended the following, "The sequence is verified by another analyst. This analyst verifies the autosampler sequence, port position, chem. station sequence, and Chrom worklist. The analyst annotates in the instrument maintenance log if the sequence has been verified."

19.4.13. Changed Section 11.7.4 from – 'If the default volume is 250 mL then 410 mL should be trapped' to 'If the default volume is 250 mL then 510 mL should be trapped.'

19.4.14. Combined GC operating conditions and BFB operating conditions (Attachment 2 and Attachment 3) as they are the same. Renumbered the attachments.

19.4.15. Updated Attachments 7, 8, and 10 to current versions.

19.4.16. Editorial comments.

19.4.17. Inserted Attachment 11 – GRO Analysis using Method TO-15

19.4.18. Editorial changes.

19.5. WS-MSA-0015, Revision 1.2, Effective 10/25/2013

19.5.1. Added psig to psia conversion in section 3.10.

19.5.2. Removed reference to Costa Mesa SOP LA-QAS-002.

19.5.3. Added language to clarify “certified clean” in section 8.2.

19.5.4. Changed humidification of method blank canister from 100uL to 50uL with distilled or NanoPure water in section 9.3.

19.5.5. Added language to evaluate EICP for S/N determination in section 10.4.1.

19.5.6. Insert section 10.4.2 to state qualitative compound identification criteria must be met for all concentrations.

19.5.7. Insert 10.4.5 to add language for re-fit / read-back evaluation for % error.

19.5.8. Removed section 11.5 from previous version regarding water addition.

19.5.9. Added language in section 11.7.6 for sample dilution that can is pressurized to 24-26 psia or no more than 3x the initial pressure.

19.5.10. Added language in method modification to use UHP Nitrogen for standards preparation instead of zero air, section 17.1.

19.5.11. Added language in method modification that UHP Nitrogen is used for dilution / pressurization, section 17.1

19.5.12. Added language in method modification that leak check is performed by measuring vacuum instead of pressure as stated in the source method, section 17.3.

19.6. WS-MSA-0015, Revision 1.1, Effective 04/22/2013

19.6.1. Inserted Section 5.1 to address transfer of sample from Tedlar bags to canisters.

19.6.2. Added Section 17.11 regarding the supporting evidence for transferring samples from Tedlar bags to canisters to prolong the holding time.

19.6.3. Updated Attachment 1 to reflect the analytes evaluated at this time.

19.6.4. Removed Attachment 2 until such time as an additional mix is introduced to the analysis.

19.6.5. Section 9.2, removed references to LCS Duplicate.

19.6.6. Inserted Section 9.1.1 regarding LCS duplicates.

19.6.7. Removed all other references to LCS duplicates (aside from Section 9.1.1)

19.6.8. Replaced all references to LA-SRA-002 with WS-QA-0032.

19.7. WS-MSA-0015, Revision 1, Effective 04/08/2013

19.7.1. Revised Attachment 3 to update conditions.

19.7.2. Revised Attachment 4 to update conditions.

19.7.3. Editorial revisions.

19.8. WS-MSA-0015, Revision 0, Effective 01/18/2013

19.8.1. This is the first version of this SOP.

Attachment 1: Standard Mix Analytes and Characteristic Ions

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
1,1,1,2-Tetrafluoroethane	811-97-2	86	69, 51	1.0
1,1,1-Trichloroethane	71-55-6	97	99, 61	0.30
1,1,2,2-Tetrachloroethane	79-34-5	83	85, 131	0.40
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	101	151, 103	0.40
1,1,2-Trichloroethane	79-00-5	97	83, 85	0.40
1,1-Dichloro-1-fluuoroethane	1717-00-6	81	61, 45	0.40
1,1-Dichloroethane	75-34-3	63	65, 83	0.30
1,1-Dichloroethene	75-35-4	61	96, 98	0.80
1,1-Dichloropropene	563-58-6	75	110, 39	0.40
1,1-Difluoroethane	75-37-6	51	65	0.40
1,2,3-Trichlorobenzene	87-61-6	180	182, 145	2.0
1,2,3-Trichloropropane	96-18-4	110	97, 75	0.40
1,2,4-Trichlorobenzene	120-82-1	180	182, 145	2.0
1,2,4-Trimethylbenzene	95-63-6	120	105, 77	0.80
1,2-Dibromo-3-chloropropane	90-12-8	157	155, 75	2.0
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	135	85, 87	0.40
1,2-Dichlorobenzene	95-50-1	146	148, 111	0.40
1,2-Dichloroethane	107-06-2	62	49, 64	0.80
1,2-Dichloropropane	78-87-5	76	63, 62	0.40
1,3,5-Trimethylbenzene	108-67-8	120	105, 77	0.40
1,3-Dichlorobenzene	541-73-1	146	148, 111	0.80
1,3-Dichloropropane	142-28-9	76	41,78	0.80
1,4-Dichlorobenzene	106-46-7	146	148, 111	0.40
1,4-Difluorobenzene	540-36-3	114	88, 63	
1,4-Dioxane	123-91-1	88	58	0.80
1.1.1.2-Tetrachloroehtane	630-20-6	131	133, 95	0.40
2,2-Dichloropropane	78-87-5	77	97, 41	0.80
2-Butanone (MEK)	78-93-3	72	43, 57	0.80
2-Chlorotoluene	95-49-8	126	91, 89	0.40
2-Hexanone	591-78-6	58	43, 85100	0.40
2-Methyl-2-propanol	75-65-0	59	57, 41	2.0
3-Chloro-1-propene (Allyl Chloride)	107-05-1	41	76, 78	0.80
4-Ethyltoluene	622-96-8	120	105, 77	0.40
4-Isopropyltoluene	99-87-6	119	134, 91	0.80
4-Methyl-2-pentanone (MIBK)	108-10-1	43	58, 100	0.40
Acetone	67-64-1	43	58	5.0
Acrolein	107-02-8	56	55, 53	2.0
Alpha Methyl Styrene	98-83-9	118	103, 117	0.40
Benzene	71-43-2	78	77, 52	0.40

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
Benzyl chloride	100-44-7	91	126, 63	0.80
Bromobenzene	108-86-1	156	77, 158	0.80
Bromoform	75-25-2	173	175, 171	0.40
Bromomethane	74-83-9	94	96, 79	0.80
Butadiene (1,3-Butadiene)	106-99-0	39	54, 53	0.80
Butane	106-97-8	43	41, 58	0.40
Carbon disulfide	75-15-0	76	78, 44	0.80
Carbon tetrachloride	56-23-5	117	119, 121	0.80
Chlorobenzene	108-90-7	112	114, 77	0.30
Chlorodibromomethane	124-48-1	129	127, 131	0.40
Chlorodifluoromethane	75-45-6	51	67, 50	0.80
Chloroethane	75-00-3	64	49, 66	0.80
Chloroform	67-66-3	83	85, 47	0.30
Chloromethane	74-87-3	50	52	0.80
cis-1,2-Dichloroethene	156-59-2	96	61, 98	0.40
cis-1,3-Dichloropropene	10061-01-5	75	77, 39	0.40
Cyclohexane	110-82-7	84	56, 69	0.40
Cyclohexanone	108-94-1	55	42, 98	0.80
Dibromomethane	74-95-3	174	93, 95	0.40
Dichlorobromomethane	75-27-4	85	83, 129	0.30
Dichlorodifluoromethane	75-71-8	85	87, 50	0.40
Dichlorofluoromethane	75-43-4	67	69	0.40
Ethanol	64-17-5	46	45	1.0
Ethyl acetate	141-78-6	43	61, 70	0.30
Ethylbenzene	100-41-4	91	106, 65	0.40
Ethylene Dibromide (EDB)	106-93-4	107	109, 81	0.80
Hexachlorobutadiene	87-68-3	225	223, 227	2.0
Hexane	110-54-3	43	57, 86	0.80
Iodomethane	74-88-4	142	127	0.40
Isooctane (2,2,4-Trimethylpentane)	540-84-1	57	41, 56	0.40
Isopropyl alcohol	67-63-0	45	4359	0.80
Isopropyl ether	108-20-3	45	87, 59	0.80
Isopropylbenzene	98-82-8	120	105, 79	0.80
Methyl methacrylate	80-62-6	69	41, 100	0.80
Methyl tert-butyl ether	1634-04-4	73	57, 41	0.80
Methylene Chloride	75-09-2	49	84, 86	0.40
m-Xylene & p-Xylene	179601-23-1	91	106, 77	0.80
Naphthalene	91-20-3	128	102, 129	0.80
n-Butanol	71-36-3	56	41, 43	2.0
n-Butylbenzene	104-51-8	92	91, 134	0.40
n-Heptane	142-82-5	43	41, 100	0.80

Analytes	CAS #	Quant	Confirmation	RL (ppb v/v)
n-Nonane	111-84-2	43	57, 85	0.80
n-Octane	111-65-9	43	41, 85	0.40
N-Propylbenzene	103-65-1	91	120, 65	0.40
o-Xylene	95-47-6	91	106, 105, 78	0.40
Pentane	109-66-0	43	57, 72	0.80
Propane	74-98-6	43	42, 39	0.40
Propane, 2-methyl	75-28-5	43	41	0.40
Propene	115-07-1	41	39, 42	0.40
sec-Butylbenzene	135-98-8	105	134, 91	0.40
Styrene	100-42-5	104	78, 103	0.40
Tert-amyl methyl ether (TAME)	994-05-8	73	55, 87	0.80
Tert-butyl ethyl ether	637-92-3	59	87, 57	0.80
tert-Butylbenzene	98-06-6	91	119, 134	0.80
Tetrachloroethene	127-18-4	166	164, 131	0.40
Tetrahydrofuran	109-99-9	42	71, 72	0.80
Toluene	108-88-3	91	92, 65	0.40
trans-1,2-Dichloroethene	156-60-5	61	96, 98	0.40
trans-1,3-Dichloropropene	10061-02-6	75	77, 39	0.40
Trichloroethene	79-01-6	130	95, 132	0.40
Trichlorofluoromethane	75-69-4	101	103, 66	0.40
Vinyl acetate	108-05-4	43	86, 42	0.80
Vinyl bromide	593-60-2	106	108, 81	0.80
Vinyl chloride	75-01-4	62	64, 61	0.40
Xylenes, Total	1330-20-7	NA	NA	1.20
Internal Standards				
Chlorobromoethane	107-04-0	130	49.128	
1,4-Difluoroethane	540-36-3	114	88, 63	
Chlorobenzene-d5	3114-55-4	117	82. 54	
Surrogates				
1,2-Dichloroethane-d4	3855-82-4	85	87, 102	
Toluene-d8	2037-26-5	100	98	
4-Bromofluorobenzene	460-00-4	95	174, 176	

Attachment 2: GC Operating Conditions, BFB and EPA TO-15

Method file: TO15				
Method File List				
GC Type: 6890				
Run type: Scan, GC, E1				
Column: Cap				
Split Ratio: 40.5:1				
	Inj.P	**Intfc**	**Source**	
Temp, °C	**150°C**	**220°C**	**NA**	
GC/DIP				
Initial Column Temperature / Hold time	**35°C for 5 minutes**			
Temperature Profile	**35°C - 110°C at 6.0°C /minute, hold for 0.10 minute, 110°C - 220°C at 8.0°C, hold for 1 minute**			
Post Time	**5.0 minutes**			
Oven equilibration Time: 0 min				
Run time: 37.35 min				
Scan Start time: 3.95 min				
Scan Parameters:				
Mass Range: 35 to 300				
Multiplier voltage: varies				
Number of samples: 2				
Threshold: 200 counts				

Attachment 3: BFB Acceptance Criteria, EPA TO-14A

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0 to 9.0% of mass 174
176	>95% but <101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 4: BFB Acceptance Criteria, EPA TO-15

Mass	Ion Abundance Criteria
50	8.0 to 40% of mass 95
75	30 to 66% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2% of mass 174
174	50 to 120% of mass 95
175	4.0 to 9.0% of mass 174
176	93% to 101% of mass 174
177	5.0 to 9.0% of mass 176

Attachment 5: Internal Standards

Bromochloromethane
1,4-Difluorobenzene
Chlorobenzene-d5

Attachment 6: Surrogate Standards

1,2-Dichloroethane-d4
Toluene-d8
4-Bromofluorobenzene

Attachment 7: Example GC/MS Initial Calibration Curve Review Checklist

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Sacramento

ICAL Review Checklist
Volatile Air GCMS

Method & Instrument ID: _____ **ICAL Date:** _____
Chrom Worklist #: _____ **Calibration ID:** _____ **TALS Analysis Batch:** _____
Method (Check the applicable box)
☐ TO-14A / TO-15 Modified SCAN ☐ TO-15 Scan ☐ TO-15 SIM ☐ TO-15 SIM (Special Project)

Acceptance criteria are found in the applicable laboratory SOP or in TALS. If item is No, create an NCM.

Review Items	Level 1		NA	Level 2
General Criteria	Yes	No		
Standards analyzed within 24 hour tune time.				
Chrom Peak Review calibration reports (F6) reviewed for error flags				
Retention time correct for isomers/coeluters and all other analytes: • Dichlorodifluoromethane / 1,2-Ddichlorofluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorofluoroethane • Hexane / Vinyl acetate • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropane / trans-1,3-Dichloropropene • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlorobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzne / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Curve reviewed in Chrom for all analytes and all appropriate leves are included/excluded				
Uploaded all calibration levels to TALS after any changes were made in Peak Review				
Method for this worklist set as Most Recent Method				
ICAL locked in Chrom and in TALS				
ICV passes -See criteria below				
Manual Integrations clearly identified, initialed, dated and reason given.				
SIM Criteria				
PFTBA tune documentation present in Chrom & TALS and meets criteria				
RSD < 30% (≤ 40% for up to 2 analytes.				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 5 points curve).				
All internal standards are within 60-140% of ICAL midpoint				
ICV (Second Source) meets 70-130% criteria (60-140%) for up to 2 analytes.				
Scan Criteria				
BFB Tune documentation present in Chrom & TALS and meets criteria				
For GRO: WS-GRO report (F8) reviewed to ensure IS/Surrogate subtraction from area sum.				
RSD < 30% (≤ 40% for up to 2 analytes, and ≤ 55% for up to 4 poor performers defined in SOP)				
Linear regressions >0.995 and intercept <+/- ½ RL/IS amount, (minimum of 6 points curve).				
All internal standards are within 60-140% of ICAL midpoint (for TO-15 / TO-15 Modified) or within 50-200% of ICAL midpoint (for TO-14A)				
ICV (Second Source) meets 70-130% criteria (60-140% for up to 2 analytes, and 45-155% for up to 4 poor performers defined in SOP)				

	Repeat Failure from Prev ICAL	
ICAL compounds outside RSD criteria (write compound and %RSD _____	Yes	No
ICV (second source) compounds outside RSD criteria (write compound and %D _____	Yes	No

1st Level Reviewer: _____ Date: _____
2nd Level Reviewer: _____ Date: _____
Comments: _____

Attachment 8: Example GC/MS Technical Data Review Checklist

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Sacramento

MS VOA AIR Data Review Checklist

Job(s):_____ Instrument ID: _____

☐TO-14A ☐TO-14A MOD ☐TO-15 ☐TO-15 MOD ☐TO-15 SIM

ICAL Batch: _____ TALS Batch:_____

Review Items	Level 1 Yes	Level 1 No	Level 2	NA
Initial Calibration				
1. Is ICAL locked in Chrom & TALS?				
2. Is ICV properly linked in TALS for L3/L4 jobs?				
Continuing Calibration				
1. BFB tune documentation meets criteria? The PFTBA Tune Documentation is attached in TALS (SIM methods only)?				
2. All Internal Standards within 50-200% of ICAL mid-point (TO-14A/TO-14A MOD) or 60-140% (TO-15/ TO-15 MOD)?				
3. Does the %D meet:30%D for standard program, and 20%D for DoD?				
4. Isomeric pairs checked for correct peak assignment? • Dichlorodifluoromethane / 1,2-Dichlorofluoroethane • Trichlorofluoromethane / 1,1,2-Trichlorofluoroethane • 2-Methyl butane / Acrolein • 1,1-Dichloroethene / cis-1,2-Dichloroethene / trans-1,2-Dichloroethene • Hexane / Vinyl acetate • 1,2-Dichloroethane / Benzene • cis-1,3-Dichloropropane / trans-1,3-Dichloropropene • Ethylbenzene / m/p-Xylene / o-Xylene • 4-Ethyl toluene / 1,3,5-Trimethylbenzene / 1,2,4-Trimethylbenzene • tert-Butylbenzene / 4-Isopropyltoluene • 1,3-Dichlorobenzene / 1,4-Dichlorobenzene / 1,2-Dichlorobenzene • 1,2,4,5-tetramethylbenzene / 1,2,3,4-tetramethylbenzene • 1,2,4-Trichlorobenzene / 1,2,3-Trichlorobenzene				
Client Samples & QC Sample Results				
1. Was analysis done within holding times?				
2. Are Chromatograms reviewed and spectra verified?				
3. Are positive results within calibration range?				
4. Are all positive results within RT windows?				
5. All target cpds in MB < RL (< ½ RL for DoD)? Requires NCM if "no."				
6. Are surrogate recoveries for MB and LCS/LCSD within control limits?				
7. Are target constituents in LCS/LCSD within control limits?				
8. Are all Chrom graphics uploaded?				
9. Are all QC samples properly linked in TALS?				
10. Do the Internal Standards meet criteria +/-50% to +/-200% (TO-14A / TO-14A MOD), +/-60% to +/- 140% (TO-15/TO-15 MOD)?				
11. Do results (e.g., dilutions) make sense?				
12. Are MS/MSD recoveries and RPDs within control limits?				
13. Have all flags been reviewed for appropriateness?				
14. All manual integrations appropriate and documented?				
15. Are non-conformances documented as NCMs?				
16. Are client sample surrogate failures flagged and NCM written?				
17. Dilutions due to target cpds? ____ Dilutions due to non-targets? ____				
18. Has QC checker been run?				

1st Level Reviewer / Date:_____ 2nd Level Reviewer / Date: _____

Comments: _____

c:\forms\checklists\msa-002 air gcms data review 2014-02-24.doc MSA-002 RKE 07/16/2014

Attachment 9: Schedule for Routine Maintenance of Analytical Instrument

Frequency	Maintenance Item
Daily	Check baseline level with analysis of blanks. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: Check for proper operation. Leak check system.
As needed	Replace septum. Clean injector port. Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add or change oil, if needed. Replace the exhaust filters on the mechanical rough pump every 1-3 years, or as needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Check ion source and analyzer (clean, replace parts as needed). Clean source, including all ceramics and lenses - as is indicated by a variety of symptoms including inability to properly tune, poor response, and high background contamination. Repair/replace jet separator. Replace filaments when burnt out or performance indicates need for replacement.

Attachment 10: Canister Pressurization Logbook (Example Page)

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Sacramento

Canister Pressurization Log

Date	Sample ID	Can #	Initial Press. (psig)	Initial Press. (psia)	Final Press. (psig)	Final Press. (psia)	Gauge ID	Initial	Comments
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		
							A B		

A = Gauge CD5437; B = Gauge _____

[Calculation : psig + 14.7 = psia]

PAGE 1

Reviewed by /Date: _____

QA.8131 GEC 7/21/2014

1. SCOPE AND APPLICATTION

1.1. This method describes the analysis of volatile petroleum hydrocarbons. The procedure used for the petroleum hydrocarbons is based on SW-846. Refer to Table A1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 218°C. Classes of compounds best suited for evaluation by this analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

2. SUMMARY OF METHOD

Refer to Section 2 of the main body of this SOP for a summary of the method.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of the method.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for Safety Information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for equipment and supplies.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Reagents

7.1.1. UHP Helium for carrier gas

7.1.2. UHP H_2 for detector combustion

7.1.3. Zero-grade air for detector combustion

7.1.4. UHP N_2 for sample preparation, standard preparation, and method blanks

7.1.5. Liquid N_2

7.2. Standards – Neat standards are good for 1 year from date purchased or if a vendor supplied standard has an earlier expiration date then that date is used.

7.2.1. Certified BTEX, MTBE, and alkane standards in pressurized gas cylinders and neat liquids, obtained from select NIST-approved vendors.

7.2.2. Certified Hexane standards in pressurized gas cylinder and neat liquid, obtained from select NIST-approved vendors.

7.2.3. Certified neat unleaded gasoline standards, obtained from select NIST-approved vendors.

7.3. Standard Preparation

Standard preparation activities are performed accordance with TestAmerica SOP WS-QA-0017.

7.3.1. Dilutions of standards are made on a volume/volume basis using serial dilution methodologies. UHP N_2 is used as the diluent gas.

7.3.2. Primary gas standards are either purchased from manufacturers in pressurized, pre-mixed gas cylinders, or prepared in passivated canisters or air sample bags from neat liquid standards.

7.3.2.1. Primary Gas Standard Prepared in Canisters - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected into a clean and evacuated (-30 inches of mercury) passivated canister. The density of the compound is used to determine the mass injected. The canister is pressurized using UHP N_2 and is then allowed to equilibrate.

7.3.2.2. Primary Gas Standard Prepared in Air Sample Bags - To prepare a primary gas standard from neat liquids, an aliquot of the compound is injected with a volumetric gas-tight syringe into a new and clean air sample bag. The air sample bag is filled with UHP N_2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

To calculate the concentration of the standard prepared:

$$ppmv = \frac{\left(D \times V_1 \times 24.45 \times 10^6\right)}{\left(MW \times V_2\right)}$$

Where:

V_1 = aliquot of neat liquid (mL)

D = density of neat liquid (g/mL)

V_2 = final volume in passivated canister or air sample bag (L)

24.45 = molar volume (L/mole) at STP

10^6 = conversion factor

MW = molecular weight (g/mole)

7.3.3. Working gas standards are good for 6 months from date of preparation. They are prepared in passivated canisters or air sample bags by making dilutions of the primary pre-mixed gas cylinders (from vendors) or by making dilutions of the prepared primary gas standard prepared from the neat liquid standards (see Section 7.3.2).

7.3.3.1. Working Gas Standard Prepared in Canisters - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a clean and evacuated passivated canister. The aliquot is metered in by measuring the vacuum of the canister as the aliquot is being transferred. The ratio of the final canister pressure and the pressure transferred is the dilution factor. The canister is pressurized with UHP N_2 and is then allowed to equilibrate.

7.3.3.2. Working Gas Standard Prepared in Air Sample Bags - To prepare a working gas standard, an aliquot of the prepared primary gas standard or of the pre-mixed primary gas standard is transferred to a new and clean air sample bag. The aliquot is transferred by direct injection with a volumetric gas-tight syringe. The air sample bag is filled with UHP N_2 to the appropriate volume using a volumetric gas-tight syringe. The air sample bag standard is then allowed to equilibrate.

7.3.3.3. To calculate the concentration of the standard prepared:

$$ppmv = C_{ps} \times {P_i}/{P_f}$$

Where:

C_{ps} = concentration of compound in primary gas standard, ppmv

P_i = aliquot of primary gas standard used in psia or in mL if using air sample bag

P_f = final pressure of canister in psia or final volume in mL if using air sample bag

7.3.4. Gasoline Calibration Standards

7.3.4.1. The primary standards are prepared in passivated canisters from neat unleaded gasoline. Gasoline from one vendor will be used to prepare two or more gasoline calibration standards. The second

source LCS is prepared from unleaded gasoline obtained from a different lot number.

7.3.4.2. A 10-, 25-, 50-, or 100-µL gas-tight syringe is used to transfer the neat gasoline into the septum-capped evacuated canister. The total (barrel + needle) syringe volume is accounted for.

8. SAMPLE PRESERVATION AND STORAGE

Refer to Section 8 of the main body of this SOP for information regarding sample preservation and storage.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the main body of this SOP for general quality control requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 30%. If an analyte exceeds the criteria, then a regression line (linear or curved) may be attempted.

10.1.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the **normal** concentration/response behavior of the detector.

10.1.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is ≤ 30%. The equation for average response factor is:

Equation A1

$$\textit{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.1.3. Linear regression

The linear fit uses the following function:

Equation A2

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response

x = Concentration

a = Slope

b = Intercept

10.1.4. Quadratic curve

The quadratic curve uses the following function:

Equation A3

$$x = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where: x = concentration

y = instrument response

a = 2nd order coefficient (curvature)

b = 1st order coefficient (slope)

c = constant (intercept)

10.2. Evaluation of calibration curves

The %RSD from the calibration curve is used to evaluate the initial calibration. If a regression line is used, the coefficient of determination (r2) shall be greater than or equal to 0.990, or the correlation coefficient (r) shall be greater than or equal to 0.995, whichever is appropriate to the regression fit used.

10.3. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response should be less than ± ½ the reporting limit for the analyte.
- The RSD for average response factors for each component must be ≤ 20%.
- When RSD is inappropriate, either the correlation coefficient (r) must be ≥ 0.995, or the coefficient of determination (r^2) must be ≥ 0.990.

10.4. Initial Calibration (ICAL)

10.4.1. At the beginning of each initial calibration curve for gasoline or other hydrocarbon fuel mixtures, the carbon range is established from the n-alkanes in the calibration standard. The start and end points for the peak summing range is the retention time at the apex of the beginning and ending carbon

range of the fuel mixture (e.g RT of C6 to RT of C12 measured at the beginning of the start peak and the end of the end peak.

10.4.2. The initial calibration for the GRO curve consists of 6 calibration standards (concentrations listed in Table A3)

10.5. Quantitating hydrocarbon mixtures:

Starting and ending retention times for quantitation are determined for each fuel as per 10.4.1. The peak areas between the starting and ending times are summed and 1,4-Difluorobenzene is used to generate a response factor. This factor is used to quantitate sample results, and depending on the client requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.6. Integration

10.6.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.6.1.1. The integrated peaks should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.6.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.7. Evaluate the initial calibration as per 10.1 and 10.3.

10.8. A second source standard is analyzed immediately following the initial calibration. The acceptance criteria for the second source standard is ± 30% from the expected value.

10.9. Continuing Calibration Verification (CCV)

10.9.1. A CCV (5000ppbv or another mid-level standard) is analyzed at the beginning of each 24 hour analytical sequence. The integration for the GRO CCV follows the same technique as stated in 10.6.

10.9.2. The acceptance criteria for the CCV is ± 30% D.

11. PROCEDURE

11.1. Refer to Section 11 of the main body of this SOP for information regarding sample preparation and other procedures.

12. CALCULATIONS/DATA REDUCTION

12.1. Refer to Section 12 of the main body of this SOP for general information regarding data analysis and calculations.

12.2. Gasoline is quantitated using the area between two marker peaks as specified in Table A2.

12.3. Calculate results in samples as in Section 12 of the main portion of this SOP.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance criteria.

14. POLLUTION CONTROL

Refer to Section 14 of the main body of this SOP for information relating to pollution control.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for information relating to waste management.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035, 8000B, and 8015B.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, March 2003, Section 8000C.

16.3. Leaking Underground Fuel Tank Field Manual, State of California, October, 1989

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. This method is performed on a GC/MS, with surrogate values calculated using Method TO-14A/TO-15, and the TPH values calculated using the total area chromatogram. All of the reference methods are based on GC-FID as the detector and the limitations thereof.

18. ATTACHMENTS

18.1. Table A1 – Standard Analyte List

18.2. Table A2 – Volatile Petroleum Hydrocarbon Quantitation Ranges.

18.3. Table A3 - Calibration Standard Concentrations

18.4. Table A4 - LCS Spiking

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Table A1		
Standard Analyte List		
Compound	CAS Number	Reporting Limit (ppbv/v)
Gasoline Range Organics (GRO)	NA	100

Table A2		
Volatile Petroleum Hydrocarbon Quantitation Ranges.		
Regulatory Method GRO	Quantitation Range	
	Start	End
GRO	n-C3	n-C12
nC4-nC12	nC4	nC12
C4-C12	n-C4	n-C12

[1] *The method specifies that the peak summing window must start at the start of the n-C6 peak and finish at the start of the n-C10 peak.*

Table A3						
Calibration Standard Concentrations (ppbv/v)						
Component	1	2	3	4	5	6
Gasoline	100	500	1000[1]	5,000	10,000	20,000

[1]*Designates the mid-level standard concentration used for the ICV and CCVs following the initial CCV of a sequence.*

Refer to the main body of this SOP for surrogate details.

Table A4	
LCS Spiking	
Component	Concentration (ppbv)
GRO	5000