Naval Facilities Engineering Systems Command Hawaii
JBPHH HI

Final

WORK PLAN

CONCRETE TANK REMOVAL RED HILL BULK FUEL STORAGE FACILITY

JOINT BASE PEARL HARBOR-HICKAM, OAHU, HAWAII

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August 2022

Prepared for Naval Facilities Engineering Systems Command Pacific by:
Cape Environmental Management Inc
155 Kapalulu Place, Suite 111
Honolulu, HI 96819

Contract Number: N62742-16-D-1807
Task Order Number: N6274222F0135

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Acronyms and Abbreviations

°C ..... degrees Celsius
°F ..... degrees Fahrenheit
ACOR ..... Alternate Contracting Officer's Representative
AGE ..... Advanced Geotechnical Engineering, LLC
AHA ..... Activity Hazard Analysis
amsl ..... above mean sea level
APP ..... Accident Prevention Plan
AST ..... aboveground storage tank
bls ..... below land surface
BMP ..... Best Management Practice
BTEX ..... benzene, toluene, ethylbenzene and xylene
BWS ..... Board of Water Supply
CAPE ..... Cape Environmental Management Inc
CFR ..... Code of Federal Regulations
COC ..... Chain of Custody
COR ..... Contracting Officer's Representative
CRZ ..... chemical reduction zone
CQCSM ..... Construction Quality Control Systems Manager
CTO ..... Contract Task Order
cy ..... cubic yard(s)
dBA ..... decibels on an A-weighted scale
DoD ..... Department of Defense
DON ..... Department of the Navy
DQCR ..... Daily Quality Control Report
DRO ..... Diesel Range Organics
DTSC ..... Department of Toxic Substances Control
DU ..... Decision Unit
EAL ..... Environmental Action Level
ECATTS ..... Environmental Compliance Assessment, Training, and Tracking System
ECEPP ..... Environmental Conditions/Environmental Protection Plan
EDS ..... Environmental Data Services
EM 385-1-1 ..... USACE Safety and Health Requirements Manual
EPA ..... Environmental Protection Agency
EZ ..... exclusion zone
FCR ..... Field Change Request
FEAD ..... Facility Engineering and Acquisition Division
FMD ..... Facility Management Department
ft ..... feet
GRO ..... Gasoline Range Organics
HAR ..... Hawaii Administrative Rules
HAZWOPER ..... hazardous waste operations and emergency response
HDOH ..... State of Hawaii Department of Health
HEER ..... Hazard Evaluation Emergency Response

HVAC ..... heating, ventilation, and air conditioning
IDW ..... Investigation-Derived Waste
IEPD ..... Installation Environmental Program Director
JBPHH ..... Joint Base Pearl Harbor-Hickam
JP-5 ..... Jet Propulsion-5
KO ..... Contracting Officer
LOD ..... Limit of Detection
LOQ ..... Limit of Quantitation
MDL ..... Method Detection Limit
mg/kg ..... milligram(s) per Kilograms
MI ..... multi-increment
NAVFAC ..... Naval Facilities Engineering Systems Command
NAVREGHI ..... Navy Region Hawaii
NELAP ..... National Environmental Laboratory Accreditation Program
NIOSH ..... National Institute for Occupational Safety and Health
NTR ..... Navy Technical Representative
NPDES ..... National Pollutant Discharge Elimination System
ORO ..... Oil Range Organics
OSHA ..... Occupational Safety and Health Administration
PAH ..... polycyclic aromatic hydrocarbon
PCS ..... Pacific Commercial Services
PGM ..... Program Manager
PID ..... photoionization detector
PM ..... Project Manager
POC ..... Point of Contact
PPE ..... Personal Protective Equipment
PQCP ..... Project Quality Control Plan
PVT Landfill ..... PVT Land Company, LTD
PWS ..... Performance Work Statement
QA ..... Quality Assurance
QC ..... Quality Control
QCO ..... Quality Control Officer
QSM ..... Quality Systems Manual
RCRA ..... Resource Conservation and Recovery Act
RDC ..... Regional Dispatch Center
RPM ..... Remedial Project Manager
S&H ..... safety and health
SAP ..... Sampling and Analysis Plan
SPCC ..... Spill, Prevention, Control, and Countermeasure
SS ..... Site Superintendent
SSHO ..... Site Safety and Health Officer
SSHP ..... Site Safety and Health Plan
SVOC ..... semi-volatile organic compound
SZ ..... support zone
TBD ..... to be determined
TGM ..... Technical Guidance Manual

TPH ..... Total Petroleum Hydrocarbons
TPH-d ..... Total Petroleum Hydrocarbons as diesel
TPH-g ..... Total Petroleum Hydrocarbons as gasoline
UIC ..... Underground Injection Control
USACE ..... United States Army Corps of Engineers
USDA SCS ..... United States Department of Agriculture, Soil Conservation Service
USFWS ..... United States Fish and Wildlife Service
VOC ..... volatile organic compound
WMP ..... Waste Management Plan
WP ..... Work Plan

1.0 Introduction

This Work Plan (WP) has been prepared for the Naval Facilities Engineering Systems Command Hawaii (NAVFAC Hawaii) under Contract Number N62742-16-D-1807, Contract Task Order (CTO) Number N6274222F0135 by Cape Environmental Management Inc (CAPE). The project area is the Red Hill Bulk Storage Facility, Joint Base Pearl Harbor-Hickam (JBPHH), Oahu, Hawaii. The area of the concrete holding tank and leach tank is referred to as the Site and Facility.

All work will be performed in accordance with all applicable State and local laws, regulations, guidance, and policies, including State of Hawaii Department of Health (HDOH) Hawaii Administrative Rules (HAR) Chapter 11-281 and HDOH Hazard Evaluation Emergency Response (HEER) Technical Guidance Manual (TGM). In addition, this project will be completed in accordance with applicable sections of the federal regulations, including Title 29 of the Code of Federal Regulations (CFR) describing Occupational Safety and Health Standards. This WP incorporates a Waste Management Plan (WMP) included in Section 7.0, a Sampling and Analysis Plan (SAP) in Section 8.0, an Environmental Conditions/Environmental Protection Plan (ECEPP) in Section 9.0, and a Project Quality Control Plan (PQCP) included in Section 10.0. An Accident Prevention Plan (APP)/ Site Safety and Health Plan (SSHP) has been prepared under separate cover.

1.1 Purpose

The purpose of this project is to provide all services, equipment, labor, and material required to remove a concrete holding tank, a concrete leach tank, connection piping, and surrounding soil at the Red Hill Bulk Storage Facility (Figure 1) identified as potentially contaminated. Contamination is defined as the presence of petroleum, semi-volatile organic compounds (SVOCs) or volatile organic compounds (VOCs) associated with petroleum that exceed Hawaii environmental action levels (EALs).

Round 1 of excavation only involves the removal of the two concrete tanks at this location along with the removal of the connection piping. Since the removal of these tanks will not impact any existing structures such as foundations or footings, they can be removed safely without impacting any existing or removal-based equipment and vehicle staging.

Round 2 of excavation involves the removal of approximately 1,444 cubic yards (cy) of petroleum impacted soil to a depth of 30 feet below land surface (ft bls), and an additional approximate 508 cy associated with a layback area necessary for slope stability during construction. Therefore, the total expected quantity to be excavated is 1,952 cy.

1.2 Scope of Work

The project objective will be accomplished by providing materials and services to excavate, remove, transport, and dispose of two concrete tanks, associated connection piping, and surrounding grossly-contaminated soil. In addition, the project includes pre-excavation waste characterization, post excavation soil confirmation sampling and testing, and site restoration.

The scope of work elements are summarized below and discussed in greater detail in subsequent sections of this WP:

- **Permitting and Planning:** Prior to mobilization to the Site, all project planning and support documents (e.g., this WP and associated APP/SSHP) will be finalized and submitted to the DON for approval, and all appropriate agencies will be notified of the intended fieldwork start date. A pre-construction meeting will also be conducted prior to any field activities. Security pass requests will be submitted to the installation for contractor and subcontractor personnel. A subsurface operations permit will also be obtained.
- **Mobilization and Site Preparation:** All personnel and equipment needed to excavate and remove the tanks and manage the Site will be mobilized once pre-construction submittals are approved. CAPE will install orange safety fence, caution tape, and/or warning signs, as necessary, to delineate the work area and protect surrounding personnel from the activities. Coordination with the installation will be conducted to ensure that the location of the loading equipment and haul routes will not impact installation activities. CAPE will provide drinking water, sanitation, and shade facilities for on-site personnel.
- **Waste Characterization Sampling:** Waste characterization samples of soil will be collected prior to excavation.
- **Round 1 Excavation and Tank/Piping Removal**: CAPE will excavate, remove, transport, and properly dispose of the concrete holding tank, leach tank, and cast-iron connection piping. The holding tank influent pipe will be disconnected and capped.
- **Round 2 Excavation:** CAPE will excavate a total of approximately 1,952 cy of grossly-contaminated soil in the vicinity of the holding and leach tanks, including layback areas necessary to achieve the final excavation depth. This material will be transported off-site for proper disposal as non-hazardous waste.
- **Truck Sampling:** CAPE will coordinate with another contractor for MI sampling of contaminated soil being loaded into trucks for disposal. Truck sampling procedures are described in the *Final Sampling and Analysis Plan for Quantifying Total Petroleum Hydrocarbons Mass*, included as Appendix G.
- **Round 1 and Round 2 Confirmation Sampling:** Confirmation samples will be collected during and following excavation via multi-incremental sampling to confirm that groundwater protection EALs (HDOH 2017b) have been met and the action is complete.
- **Transportation and Disposal:** CAPE will remove, transport, and dispose of all residual product, sludge, grossly contaminated soil, concrete, connection pipe, and other debris. Additional materials such as residual petroleum products, sediment, and tar paper may also be present and are to be properly characterized for waste disposal. CAPE is responsible for disposal of all waste at a permitted private or non-Navy landfill.
- **Backfill and Restoration:** For the Round 1 excavation, prior to placing backfill material, CAPE will isolate native soil from backfill material using plastic sheeting or other suitable material, such as geomembrane. During Round 2 activities, the excavation will be backfilled directly using imported fill. The site and staging areas will be restored by CAPE in accordance with JBPHH Environmental requirements.
- **Demobilization:** Following completion of all the above-mentioned tasks, all construction equipment and facilities will be demobilized from the Site. CAPE will execute cleanup activities during progress of the work and at the completion of the work to keep the Site and adjacent properties free from accumulation of waste materials, rubbish, and windblown debris generated by contractor activities. A Closure Report will be prepared by CAPE summarizing all fieldwork activities.

The provisions of this WP are applicable to all CAPE employees and subcontractors engaged in this contract, and any individual or agent acting as a vendor to CAPE to provide support services and/or commodities. Certain portions of the WP apply to those personnel who might have dealings with CAPE site personnel from a safety and health (S&H) standpoint and may visit the Site. Certain provisions will be applicable to NAVFAC Hawaii, primarily safety precautions for emergency notifications in the event of an accident or incident. Work conducted under this plan will be performed in accordance with all applicable federal, state, and local safety and occupational health laws and regulations.

2.0 Site Description

2.1 Site Location and Background

The Red Hill Bulk Storage Facility is located north of the Interstate H-201, approximately 2 miles east of Pearl Harbor, as shown in Figure 1. Preservation land is located east and northeast of the Facility boundary. To the southeast are residential single-family homes in Moanalua Valley; a high cliff face with a 100–200-ft elevation difference exists between the Facility and this residential area. Southwest of the bulk fuel storage facility area on the lower southwest flank of Red Hill are the public Red Hill Elementary School and residential apartments, and further west is U.S. Coast Guard Housing on F-1 Military land. North of the western segment of the Facility boundary in South Hālawa Valley is the State Animal Quarantine Station, private businesses in Hālawa Industrial Park, and the State-operated Hālawa Correctional Facility. To the north of the Correctional Facility at the lower reaches of an intervalley ridge that forms the north wall of South Hālawa Valley is the open-pit Hālawa Quarry operated by the Hawaiian Cement Company.

On December 18, 2021, a concrete holding tank and connected concrete leach tank were identified as possibly being impacted by a Jet Propulsion-5 (JP-5) spill. The release of an unknown quantity of JP-5 fuel in the Adit 3 tunnel of the Red Hill Facility was identified on November 20, 2021. The purpose of these tanks is to discharge storm water collected in a sump located [redacted] Both cylindrical tanks are 8 ft tall, 7 ft in diameter, and tank walls are approximately 6-inches thick. Upon inspection of the holding tank, approximately 1,500 gallons of fuel/water mixture was identified. This fuel/water mixture was recovered and measured to contain approximately 253 gallons of free product and approximately 1,250 gallons of water. The leach tank was empty and completely dry; however, noticeable petroleum odors were observed (DON 2022a).

Initial site characterization was performed between January 11 and 13, 2022, by using direct-push drilling to collect soil and soil vapor readings from 22 borings. Lateral extent of contamination extends between 10 and 17.5 ft from the tanks; however, the vertical bound of contamination was not reached. A phase 2 site characterization was performed between March 9 and 17, 2022 to provide better information about the extent of contamination and whether the underlying perched aquifer has been impacted by petroleum. Based on the Phase 2 data provided in the Draft Final Technical Memorandum, Phase 2 Holding Tank and Leach Tank Characterization, November 2021 Pipeline Release (DON 2022b), the majority of the petroleum-contaminated soil appears to be within a 50-ft by 23-ft area, at an average depth of 20 ft. The data also confirmed that TPH-d, TPH-g, naphthalene, 1-methylnaphthalene and 2-methylnaphthalene exceeded the Drinking Water toxicity EALs for the shallow perched aquifer found between 28 to 31 ft bls.

2.2 Climate

Climatological conditions in the area of the concrete tanks consist of warm to moderate temperatures and low to moderate rainfall. The average annual precipitation is approximately 40 inches, which occurs mainly between November and April. Average temperatures range from the low 60s to high 80s degrees Fahrenheit (°F) (DON, 2022a).

2.3 Soils/Geology

The concrete tanks are located within the Koʻolau Volcanic series. The Koʻolau formation at Red Hill consists of the basaltic lava flows that erupted from a fissure line approaching 30 miles in length and trending in a northwest rift zone.

Pāhoehoe and aʻā lava flows are present in the Koʻolau formation. The valleys on either side of Red Hill ridge were formed as a result of fluvial erosion and are filled with sedimentary deposits (alluvium and colluvium), also known as valley fill, underlain by residual (weathered basalt), also known as saprolite. Saprolite zones in Hawaiʻi are typically around 75-ft thick but can be 300-ft thick or greater beneath the valley floors or in areas of high precipitation. The results of a 2018 seismic survey in North and South Hālawa Valleys, Red Hill, and Moanalua Valley (DON, 2018a) found that valley fill and saprolite extend much deeper in the valleys surrounding Red Hill, particularly in the center of the valleys and below the streambeds.

Soils in the vicinity of the tanks are mapped as Helemano-Wahiawā association consisting of well-drained, moderately fine-textured and fine-textured soils (United States Department of Agriculture, Soil Conservation Service [USDA SCS], 1972). The surfaces of the basaltic flows have been weathered to form reddish-brown clayey silt, which is the basis for the local name "Red Hill." These soils typically range from nearly level to moderately-sloping and occur in broad areas dissected by very steep gulches. They formed in material weathered from basalt to a depth of approximately 10 ft bls. Along the slopes, the basaltic bedrock is covered with approximately 10–30 ft of Koʻolau residuum. These soils were derived from weathering of the underlying basalt bedrock or were deposited as alluvium/colluvium. The younger alluvium/colluvium deposits were derived from fractured basalts and tuff. Beneath the surficial soils, alternating layers of clay and basalts are encountered at depth. The northwestern slope of Red Hill is generally barren of soil and consists of outcropping basalt lava flows to the valley floor (DON, 2022a).

2.4 Surface Water

Surface water features in the general vicinity of the holding tank and leach tank include South Hālawa Stream (an ephemeral, concrete lined stream approximately 70 ft to the west northwest). Potential recharge (run-on and operational water use) from the Hālawa Quarry north of the bulk fuel storage facility may also impact groundwater flow in this area. In Hālawa Valley, stream flow may contribute water to perched groundwater within alluvial material (valley fill) but is generally isolated from the underlying basal aquifer. Most precipitation percolates to the basal aquifer and does not maintain base flows in the streams. Groundwater that flows beneath the Facility does not intercept surface water inland of the ocean shoreline (DON, 2007). Both South Hālawa Stream and Moanalua Stream (to the north and south of Red Hill ridge, respectively) are located approximately 170 ft or more above the basal water table.

2.5 Groundwater

In the vicinity of Red Hill, the basal aquifer water table lies between 15 and 20 ft above mean sea level (amsl), and regionally groundwater flows toward Pearl Harbor (mauka to makai), although

potential exists for variability in localized flow directions depending on geologic formations and other factors.

The Facility, including the holding tank and leach tank area of concern, is located at the administrative boundary between the Waimalu Aquifer System of the Pearl Harbor Aquifer Sector and the Moanalua Aquifer System of the Honolulu Aquifer Sector. The underlying aquifer is classified as a basal, unconfined, flank-type aquifer and is currently used as a drinking water source.

The holding tank and leach tank area is located upgradient of the Hawaii State Underground Injection Control (UIC) Line, which separates potable groundwater from non-potable groundwater. The nearest drinking water supply well is Navy Supply Well [redacted] (also known as Red Hill Shaft), located approximately [redacted]. The nearest Honolulu Board of Water Supply (BWS) public drinking water supply well (BWS Hālawa Shaft Well [redacted]) is located hydraulically cross-gradient of the Facility, approximately [redacted] pumps water from the basal aquifer (DON, 2022a).

Perched groundwater present on Oahu is known to occur in four distinct zones: (1) water confined by intrusive rocks, (2) water perched on ash or tuff beds, (3) water perched on soil beds, and (4) water perched on alluvium. Perched groundwater in the vicinity has been found between 30 and 35 ft bls and again at 50 to 55 ft bls (Sterns and Vaksvik, 2001; DON, 2019a).

2.6 Historical Land Use

Prior to the 1940s, the surface of Red Hill supported cane sugar and pineapple agriculture. DON archive images show that the Red Hill ground surface was exposed and modified during construction of the bulk fuel storage facility beginning in 1940. A 1952 aerial photograph shows unmaintained land on the ridge of Red Hill and agriculture on the lower reaches of Red Hill north of the Moanalua Golf Course (DON, 2019b).

2.7 Current Land Use

The Facility, including the holding tank and leach tank area, is located on land zoned by the County as a mix of F-1 Federal and Military and P-1 Restricted Preservation districts. All major structures at the Facility are located underground. Populated areas closest to the Facility are ‘Aiea to the west and Honolulu to the south and east. Honolulu is heavily urbanized and densely populated.

2.8 Previous Investigations

Previous environmental investigations at the Facility are summarized in Table 2-1.

Table 2-1: Previous Environmental Investigations Summary

Investigation Report	Summary
Technical Report (DON, 2007)	An environmental investigation and risk assessment initiated in 2004 included installation of soil vapor monitoring points in angle borings under the active fuel storage tanks, three additional groundwater monitoring wells in the lower access tunnel, a three-dimensional groundwater model, and a Tier 3 human health risk assessment.

Investigation Report	Summary
Tank 17 Removal Action Report (DON, 2008)	A limited removal action and site characterization investigation was conducted in June 2008; the report's Environmental Hazard Analysis determined that the release posed no further significant environmental hazards.
Type 1 Letter Report (DON, 2010)	A 2010 investigation re-evaluated the DON (2007) groundwater model assumptions and results and the Tier 3 risk assessment results.
Monthly Soil Vapor Monitoring Reports (DON, 2014 through present)	Soil vapor photoionization detector (PID) measurements are collected monthly under the Facility's fuel storage tanks with soil vapor monitoring points.
Tank 5 Quarterly Release Response Reports (DON, 2014 to present	In response to the 2014 fuel release from Tank 5, DON reports release response actions undertaken in the last 90 days to the HDOH.
Seismic Profiling to Map Hydrostratigraphy in the Red Hill Area (DON, 2018a)	Presented results and evaluation of nine seismic profiling transects conducted at Red Hill and in North and South Hālawa Valleys and Moanalua Valley to improve understanding of subsurface conditions that affect groundwater flow and chemical fate and transport.
Groundwater Protection and Evaluation Considerations for the Red Hill Bulk Fuel Storage Facility (DON, 2018b)	Presented an interim environmental analysis of data and an initial analysis of potential environmental risks; interim results of the groundwater flow model; and an evaluation of hypothetical release scenarios.
Conceptual Site Model (DON 2018c; 2019b)	Established a basis for evaluating chemical transport pathways and potential for exposure of human receptors to potentially impacted drinking water.
Groundwater Flow Model Report (DON, 2020a)	Refined the previous groundwater flow model to improve understanding of the direction and rate of groundwater flow within the aquifers around the Facility.
Investigation and Remediation of Releases Report (DON, 2020b)	Documented the response to the January 2014 Tank 5 release and evaluated potential remedial alternatives for that release and any potential future release.
Evaluation of Chromatograms for Understanding total petroleum hydrocarbons (TPH) Detections in Monitoring Wells (DON, 2020c)	Provided an evaluation of total petroleum hydrocarbons detections in monitoring wells to determine whether those detections are indicative of potential fuel impacts from the Facility.
Tank 5 Quarterly Release Response Report, June 2021 (DON, 2021a)	Documented the results of the first quarter release response to the May 6, 2021 Tunnel Pipeline Breach release.
Initial and Quarterly Release Response Reports, Pipeline Breach in Tunnel and Fire Suppression Drain Line *(DON 2021b; 2022a)*	Documented the results of release response efforts for the May 6, 2021 Tunnel Pipeline Breach and the November 20, 2021 Fire Suppression Drain Line releases.
Draft Final Technical Memorandum, Phase 2 Holding Tank and Leach Tank Characterization November 2021 Pipeline Release Red Hill Bulk Fuel Storage Facility (DON, 2022b)	Presented a summary of the activities, methods, field observations, pre-validation Level II data package results, and pre-validation Level IV data package results to characterize the soil around the Holding Tank and Leach Tank area of concern.

According to the 2022 Draft Final Technical Memorandum (DON, 2022b), the following evidence indicates that fuel was likely released from the holding and leach tanks into the subsurface soil beneath the tanks:

- Of the 35 subsurface soil samples analyzed for total petroleum hydrocarbons as gasoline (TPH-g), the exceedances were the leaching to groundwater EAL of 696 milligrams per

kilogram (mg/kg) in 6 (six) samples, the direct exposure EAL of 451 mg/kg in 8 (eight) samples, and the gross contamination (odor) EAL of 100 mg/kg in 13 (thirteen) samples;
- Of the 35 subsurface soil samples analyzed for total petroleum hydrocarbons as diesel (TPH-d), the exceedances were the leaching to groundwater EAL of 940 mg/kg in 7 (seven samples), the direct exposure EAL of 219 mg/kg in 13 (thirteen) samples, and the gross contamination (odor) EAL of 500 mg/kg in 9 (nine) samples;
- None of the 35 subsurface soil samples exceeded any HDOH EALs for benzene, toluene, ethylbenzene, or xylenes (BTEX) during this investigation;
- Naphthalene exceeded the leaching to groundwater EAL of 3.1 mg/kg in 3 (three) subsurface soil samples; 1-Methylnaphthalene exceeded the leaching to groundwater EAL of 0.89 mg/kg in 10 (ten) subsurface soil samples, and 2-Methylnaphthalene exceeded the leaching to groundwater EAL of 1.9 mg//kg in 6 (six) subsurface soil samples;
- In addition, the leach tank sediment sample exceeded all three soil EALs, with the highest concentrations of naphthalene (7.2 mg/kg), 1-methylnaphthalene (52 mg/kg), and 2-methylnaphthalene (74 mg/kg); and
- PID headspace measurements and subsurface soil sample results from soil borings located nearest to both tanks show the highest concentrations of TPH-g, TPH-d, and naphthalenes, with the exception at the 16 to 17 ft depth interval, which has the highest concentration of TPH-d (5,900 mg/kg).

Although there were significant TPH-g exceedances, the chromatograms revealed that most of the measured constituents were in the latter part of the TPH-g carbon ranges that could be considered to be in the TPH-d range. This result does not indicate a gasoline release. The chromatograms were reviewed by the project forensic chemist who identified the chromatographic signature as very similar to JP-5 (DON, 2022b).

A discussion of the roles of key project personnel is provided in the sections that follow. If changes to CAPE or its subcontractor key personnel are required, CAPE will discuss the proposed change(s) with the DON prior to making the change(s). The following CAPE management organization has been assembled to execute the project tasks.

3.1 Project Manager

The CAPE Project Manager (PM) is responsible for the following:

- Execution of the CTO for overall conformance to NAVFAC Hawaii requirements and specifications, with respect to technical, cost, and schedule issues;
- Review of CTO submittals to ensure contract compliance; interaction and coordination with the NAVFAC Contracting Officer (KO) or designated representatives, Contracting Officer's Representative (COR), Alternate Contracting Officer's Representative (ACOR), regulatory agencies, natural resource trustees, and other stakeholders including FEAD;
- Management of all field construction activities, including directing project staff and subcontractors in accordance with the contract requirements;
- Tracking, documenting, and reporting proposed changes to the scope of work for the project;
- Direct communication with NAVFAC Hawaii regarding project execution and accountability.
- Coordination with the project QCO to ensure compliance with standard protocols and procedures and implementation of the WP;
- Coordination with the Site Safety and Health Officer (SSHO) and NAVFAC Hawaii to ensure implementation of the APP/SSHP;
- Assigning personnel consistent with contract and CTO requirements; and

- Ensuring compliance with federal, state, and local regulations and requirements as appropriate.

3.2 Site Superintendent

The CAPE Site Superintendent (SS) will ensure that work at the Site occurs in a safe manner and in accordance with the approved project WP, PQCP, and APP/SSHP. Specifically, responsibilities will include the following:

- Serving as the primary field point-of-contact (POC);
- Field implementation of the CTO, including day-to-day operations;
- Documenting all activities in the Daily Contractor Production Report;
- Overseeing field crews and ensuring that project tasks are completed according to the performance work statement (PWS);
- Sequencing subcontractors into work areas;
- Working with the PM to plan day-to-day site activities; and
- Ensuring compliance with federal and local regulations and requirements as appropriate.

3.3 Site Safety and Health Officer

The CAPE SSHO will ensure the proper implementation of the APP/SSHP. Specifically, responsibilities will include the following:

- Oversight and enforcement of the APP/SSHP, including stop-work authority;
- Serving as the main POC for any on-site emergency; and
- Ensuring compliance with project-specified S&H requirements, federal and Occupational Safety and Health Administration (OSHA) regulations, as well as the United States Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM 385-1-1; USACE, 2014).

3.4 Quality Control Officer

The CAPE QCO will ensure the proper implementation of the PQCP. Specifically, responsibilities will include the following:

- Developing, maintaining and enforcing the PQCP, with stop-work authority for quality reasons;
- Development and delivery of Daily Quality Control Reports (DQCRs);
- Performing and maintaining three phases of control for each phase of work, to ensure that work complies with contract requirements; and
- Performing inspections, tests, and periodic quality control (QC) meetings at the Site to track and report progress.

3.5 Environmental Manager/Transportation and Disposal Coordinator

The CAPE EM/TDC will ensure the proper implementation of the project EC/EPP. Specifically, responsibilities will include the following:

- Completing applicable Department of Transportation, City and County of Honolulu, and Environmental Compliance Assessment, Training, and Tracking System (ECATTS) training modules (installation-specific or general) prior to starting respective portions of on-site work
- Coordinating contractor compliance with federal, state, local, and installation requirements
- Ensuring compliance with Stormwater Program requirements and conducting stormwater best management practice inspections
- Ensuring compliance with Hazardous Materials (storage, handling, and reporting) requirements; and coordinating any handling of regulated substances
- Ensuring waste segregation and storage compatibility requirements are met; inspecting and managing Satellite Accumulation areas; ensuring only authorized personnel add wastes to containers; ensuring Contractor personnel are trained in 40 CFR requirements in accordance with their position requirements; coordinating removal of waste containers; and maintaining the Environmental Records binder and required documentation, including environmental permits compliance and close-out
- Training personnel on the ECEPP, including spill response procedures
- Profiling and manifesting waste prior to generator signature on profiles and manifests.

3.6 Contractor

Contractor information for CAPE is as follows:

Company Name: Cape Environmental Management Inc
Address: 155 Kapalulu Place, Suite 111
Honolulu, Hawaii 96819

Project Manager: [redacted]

The site supervision, inspection, and approval of all work will be the responsibility of CAPE. CAPE will adhere to the procedures identified in the PWS and will follow the procedures, QC, and site safety protocols designated therein.

3.7 Subcontractors

Subcontractor roles are listed in Table 3-1. Subcontractors for this project are responsible for laboratory services, land surveying, tank clearing and waste disposal, utility locating, data validation, and geotechnical engineering and testing services.

4.0 Regulatory Background

4.1 Hawaii Department of Health

The HDOH is the lead regulatory agency for the Site, responsible for overseeing site investigation, assessment, and remediation activities. All work for this contract will be performed in accordance with HDOH HAR Chapter 11−281 and HDOH HEER TGM.

4.2 Resource Conservation and Recovery Act

Resource Conservation and Recovery Act (RCRA) is the primary federal law governing the disposal of solid and hazardous waste. Congress passed RCRA on 21 October 1976. The hazardous waste program, under RCRA Subtitle C, establishes a system for controlling hazardous waste from the time it is generated until its ultimate disposal. Hazardous waste is a waste with properties that make it dangerous or potentially harmful to human health or the environment. RCRA Subtitle C lists the types of hazardous wastes and presents criteria by which a waste is classified as hazardous. Non-hazardous solid waste is regulated under Subtitle D of RCRA. Regulations established under Subtitle D ban open dumping of waste and set minimum federal criteria for the operation of municipal waste and industrial waste landfills.

5.0 Pre-Construction Activities

5.1 Permits, Licenses and Notifications

Operations will be performed in compliance with all applicable federal, state, local, Department of Defense (DoD), and DON laws, regulations, and policies, including HDOH HAR Chapter 11–281 and HDOH HEER TGM. In addition, this project will be completed in accordance with applicable sections of the federal regulations, including Title 29 of the CFR describing Occupational Safety and Health Standards. No National Pollutant Discharge Elimination System (NPDES) permit is required for this project due to its size; however, CAPE will implement best management practices (BMPs) as necessary to prevent the discharge of any pollutants to the stormwater system or ocean.

A dig permit will be obtained from NAVFAC prior to the start of field work.

CAPE will suspend work and notify the DON in the event differing site conditions are encountered, such as the observation of suspected endangered species and/or the unearthing of suspected artifacts and/or human remains and/or hazardous waste.

5.2 Protection of Existing Structures and Utilities

CAPE will conduct all site activities in a manner that protects aboveground and buried utilities and structures in the area, such as electric lines located west of the excavation area and an abandoned 16-inch JP-5 pipeline. In addition, intrusive activities will not disturb or compromise the integrity of groundwater monitoring wells on the Red Hill Former Oily Waste Disposal Facility (basal aquifer Well IDs OWDFMW03A, 6A, and 7A and perched aquifer Well IDs OWDFMW03B, 6B, 7B, and 7C) or aboveground infrastructure associated with the granular activated carbon units adjacent to the work area (Figure 3).

CAPE will obtain a dig permit and identify the location of subsurface structures and utilities by notifying One Call and conducting geophysical toning. The base dig permit will be submitted in advance to allow three weeks for the base dig permit to be processed.

The dig ticket will remain active for the duration of the project and is valid for 28 calendar days from the date of its issuance. A copy of the ticket will be kept onsite for reference. If work is still ongoing past the 28th day, the ticket will be renewed prior to the 28th calendar day.

To ensure that intrusive locations were not positioned over subsurface utilities, CAPE subcontracted Hawaii Private Utility Locators for additional third-party utility clearance.

CAPE will immediately notify the COR, the assigned NAVFAC Facility FEAD CM/ET and Facilities Maintenance Department (FMD) if structural damage occurs to utilities, surface features, and/or adjacent structures during the course of this project.

5.3 Safety Requirements

The project-specific APP/SSHP for this project has been prepared under separate cover. CAPE will be responsible for the health and safety of all employees and subcontractors working at the Site and will follow the applicable reference guidance documents, including:

- USACE Safety and Health Requirements Manual, EM 385-1-1 (USACE, 2014);
- Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, National Institute for Occupational Safety and Health (NIOSH) / OSHA / United States Coast Guard / Environmental Protection Agency (EPA) (NIOSH, 1985);
- 29 CFR 1910, OSHA, General Industry Standards; and
- 29 CFR 1926, OSHA, Standards for Construction.

S&H measures will be implemented upon arrival to the Site and throughout fieldwork. Work will be performed, at a minimum, in Level D personal protective equipment (PPE). Higher levels of PPE may be used, as determined to be appropriate by the onsite SS/SSHO and will be upgraded in accordance with the CAPE Safety and Health Program. The SSHP specifies the necessary monitoring, inspection, controls, training, reporting, and communication for this project. The SSHP also includes appropriate activity hazard analyses (AHAs) for the Site. AHAs may be updated or modified as required during the course of fieldwork based on necessary changes to the planned work or conditions encountered in the field. All persons working at the Site will be required to read the APP/SSHP and sign an acknowledgement verifying their understanding of the plan prior to their participation in any onsite work. Daily safety meetings will be held each morning prior to the start of work to discuss the planned activities for the day. All site visitors and new workers will receive a safety briefing and sign-in prior to entering the Site.

5.4 Pre-Construction Submittals and Meeting

All pre-construction submittals will be accepted/approved by the DON prior to commencing field activities. Pre-construction submittals consist of the WP and APP/SSHP. Draft and Final pre-construction submittals will be submitted to the CTO COR/ACOR.

The kick-off meeting will be scheduled with the CTO COR/ACOR following approval of this WP. The Contractor shall schedule a kick-off meeting with the FEAD, NAVFAC representatives, DON subject matter experts, FMD Specialist, and PM prior to the start of fieldwork. The primary objective of this meeting is to ensure that all parties understand the project tasks, schedule, and other concerns. Other topics that typically are discussed at this meeting include:

- Scope of work;
- Daily reporting, production, safety, and QC protocol; and
- Communication, coordination channels, and POCs.

Meeting minutes will be taken by CAPE and later disseminated to all attendees.

Additionally, an environmental brief will be included in the preconstruction meeting. The brief will include the following:

- Types, quantities, and use of hazardous materials that will be brought onto the installation;

- Types and quantities of wastes/wastewater that may be generated during the contract; and
- Discussion of the results of the preconstruction survey at this time.

Prior to initiating any work on site, meet with the KO and Installation Environmental Office to discuss the proposed ECEPP. Develop a mutual understanding relative to the details of environmental protection, including measures for protecting natural and cultural resources, required reports, required permits, permit requirements (such as mitigation measures), and other measures to be taken.

5.5 Security

Access to the Red Hill Bulk Storage Facility installation is restricted and is controlled by guards or locked gates. The access route to the Site is shown in Figure 2.

CAPE will obtain security passes, as necessary, for all CAPE and subcontractor personnel and vehicles requiring access to the Site. Field activities that may impact traffic near the Site consist of trucks delivering equipment and materials, use of personal and support vehicles, and trucks transporting wastes off site and fill materials on Site.

6.0 Field Activities

The following will be performed as part of field activities. Local subcontractors will be employed where appropriate to perform portions of the tank removal activities. Additional details regarding QC, sampling, and safety activities are provided in subsequent sections of this WP or in the APP/SSHP.

6.1 Mobilization

The mobilization of equipment and personnel to the Site will be conducted once the pre-construction activities have been completed.

Applicable members of the project team (Table 3-1) will be mobilized to the Site. The Round 1 team will consist of a QCO/SSHO (dual hat), SS, and a field crew consisting of sampling personnel, equipment operators, truck drivers, and laborers. The Round 2 team will consist of a QCO/EM/TDC, (dual hat), SS, SSHO, and a field crew consisting of sampling personnel, equipment operators, truck drivers, and laborers. The following equipment and supplies are expected to be mobilized to complete the project:

- Heavy equipment – excavators, bulldozer, wheel loader, articulated haul truck, roller compactor, water truck, and tool container;
- Sampling equipment, hand and power tools;
- Traffic control signs and barricades;
- PPE;
- Hand-wash and eye-wash stations; and
- Portable toilets.

The staging plan for the Round 2 excavation activities is provided as Figure 3. A minimum 20-ft buffer from the GAC discharge lines will be maintained during excavation activities. All heavy equipment will be inspected daily for leaks, loose hydraulic hoses, etc. Any hazardous materials (fuel for equipment, etc.) brought on Site will be properly stored in secondary containment at a minimum of 15 ft from any drain inlet, storm drain, etc. The nearest drainage ditch is more than 60 ft away from the leach tank and holding tank (Figures 4 and 5). Equipment apparatus (loader buckets) that come in contact with contaminated materials will be decontaminated prior to leaving the Site. Further details of equipment decontamination procedures are discussed in the APP/SSHP.

CAPE will install a temporary orange safety fence, caution tape, and/or warning signs, as necessary, to delineate the work area and protect surrounding personnel from the activities.

6.2 Establishment of Work Zones

Specific work zones will be created near the excavations for safety and security. Support zones (SZs) will be established outside of the Exclusion Zone (EZ) and Contaminant Reduction Zone (CRZ). EZs will be demarcated using temporary orange construction fence, chain-link fence, dust fence, caution tape, and/or warning signs. All appropriate traffic control measures, including but not limited to installing barricades, will be undertaken to prevent entry of any unauthorized vehicles into any of the EZs. The CRZ will be located adjacent to the EZ. The SZ will be located as close as

practical to the EZ/CRZ. Signs and placards will be posted warning against unauthorized entry, denoting construction areas, and other notices as deemed necessary by the SSHO.

6.3 Utility Clearances

CAPE will employ a qualified subcontractor to perform a subsurface survey to identify all underground utilities and any other subsurface conveyances or anomalies prior to any intrusive earthwork. Any necessary base utility clearance forms will be completed prior to intrusive earthwork.

6.4 Waste Characterization Sampling

To eliminate the need for soil stockpiling, prior to excavation, four borings to 12 ft bls will be drilled for waste characterization. Details regarding waste characterization sampling activities can be found in the SAP in Section 8.0 of this WP. Waste characterization sampling was conducted on 4-5 May 2022 and the locations are shown in Figure 4.

6.5 Removal and Disposal of Tank Contents

Prior to the removal of any tank contents, CAPE will gauge the holding tank to confirm if liquids are present and, if so, the estimated volume. The leach tank does not have a floor, and therefore there will be no contents to empty. If necessary, subcontractor PCS will remove any tank contents using a vacuum truck and containerize them for off-site disposal. CAPE will record the amount of liquid that is removed from the holding tank. Once the contents of the holding tank are removed, the tank will be cleaned prior to demolition. PCS will follow the triple rinse process. Cleaning will entail pressure washing the interior of the tank and vacuuming the rinse water and containerizing it for off-site disposal.

The connection piping between the holding and leach tanks will be flushed with a water surfactant wash (Simple Green and water or equivalent) and allowed to drain back to the tanks. The wash water will also be removed from the tank with a vacuum truck or pneumatic pump and containerized in the vacuum truck or in 55-gallon drums, as appropriate.

6.6 Round 1 Excavation and Tank/Piping Removal

The holding tank and leach tank are each approximately 8 ft tall and 7 ft in diameter and have approximate wall thickness of 6 inches. The leach tank does not have a floor. Connection piping (assumed to be cast iron) is 6 inches in diameter and approximately 40 ft in length.

Holding tank influent pipe will be disconnected and capped. CAPE will then demolish the concrete tanks in-situ using an excavator with a multi-processor attachment. After the concrete is broken apart, CAPE will excavate the demolished tanks, connection piping, and surrounding grossly contaminated soil. The anticipated excavation limits are shown in Figure 5. CAPE personnel will visually inspect the soil removed during the excavation for evidence of staining. It is not anticipated under this work order that water saturated soils will be removed, or that dewatering will be required. Excavated soil will be directed loaded into trucks for off-site disposal.

6.7 Round 2 Excavation

During the Round 2 soil excavation activities, petroleum impacted soil in the approximate 1,300 square ft area shown in Figure 6 will be excavated to a depth of 30 ft bls or the water table, whichever is encountered first. This corresponds to approximately 1,444 cy of soil that will be removed. The 30-ft excavation depth requires the sidewalls to be sloped for access and safety purposes, corresponding to an additional 508 cy of material that will be excavated, for a total of approximately 1,952 cy. During active work hours, an excavation competent person will be on-site at all times to ensure that deep excavation activities are conducted safely in accordance with the project APP/SSHP. Personnel (other than the operator in the excavator) will not enter the excavation and construction fencing and barricades (as necessary) will be installed around the perimeter of the work area and secured at night and during weekends to prevent unauthorized access to the area.

Excavation will be conducted in phases, as described below and illustrated in Appendix A:

1. Excavation between 0 and 12 ft bls will be conducted with a hydraulic excavator. The sidewalls will be sloped at a 1:1 horizontal to vertical slope to allow for safe access to the deeper area of excavation. During the initial excavation from 0 to 12 ft bls, CAPE will construct a 12-ft wide, 25% sloped (4:1 horizontal to vertical) haul road for excavation equipment and truck access. CAPE will protect utilities and monitoring wells during this phase by demarcating them in the field, and hand-digging around them as necessary.

2. Between 12 and 30 ft bls, excavation will be conducted with the hydraulic excavator located on the working platform that was created at 12 ft bls. Excavation from 12 to 30 ft bls will be conducted with vertical sidewalls, as field conditions allow. The excavator will start at the western edge of the excavation, and progress back toward the access road. Excavation depths will be confirmed using a measuring rod affixed to the excavator, or a weighted measuring line. Excavation extents may also be field-verified with a portable global positioning system unit. Completed portions of the excavation will be partially backfilled as soon as practical, but within 4 hours to provide slope stability as shown in Appendix A. The excavation will only be left open long enough to collect sidewall and floor confirmation samples. Backfill will be staged nearby to allow for immediate backfilling. A wedge of fill will be placed in lifts and tamped into place with the bucket of the excavator, and backfilling will progress as the excavation continues. If sloughing or otherwise unsafe conditions are observed, the excavation design may be modified and additional sloping and benching conducted as necessary. Excavation will be overseen by the CAPE competent person, and a geotechnical engineer from AGE may conduct periodic site visits to observe the excavation activities.

6.8 Round 1 and Round 2 Confirmation Sampling

CAPE will conduct confirmation sampling following tank and piping (Round 1) and soil (Round 2) removal. All confirmation sampling will be conducted using multi-incremental sampling and will be conducted in accordance with the SAP (Section 8.0). Upon receipt of the analytical results, sample concentrations will be compared with the groundwater protection EALs (Table A-2, HDOH 2017b). If confirmation results are below the EALs, no further action will be required; if select EALs are

exceeded, the DON may meet with HDOH and EPA to discuss whether a risk-based corrective action is needed to manage any residual contamination left in place.

6.9 Transportation and Disposal

CAPE will dispose of all residual product, sludge, contaminated soil, concrete, connection pipe, and other debris. The on-site haul route will be established to allow ingress and egress at the northeast corner of the Site (Figure 2). Truck tires and tailgates will be inspected by CAPE's laborer(s) and brushed prior to exiting the loading area. CAPE personnel will also be on site during soil loading to manage truck traffic and ensure waste manifests are properly completed prior to any trucks leaving the Site. Prior to truck loading each day, CAPE's crew will inspect and perform preventative maintenance on the heavy equipment to minimize down time. After truck loading each day, CAPE's crew will remove/adjust existing BMPs, and prepare for the following workday.

All loads will be covered, and each truck driver will possess a signed waste manifest (or applicable bill of lading) prior to exiting the Site. Loads will be transported and disposed in accordance with the WMP. Based on the latest sampling events at the site, non-hazardous waste is anticipated to be encountered during the removal. All soil will be tested for RCRA hazardous waste toxicity characteristics to determine proper transportation and disposal requirements. If RCRA hazardous waste is identified during the pre-construction waste characterization effort, the soil will be separated from the tank and piping materials and drummed for disposal.

6.10 Backfill and Restoration

6.10.1 Round 1 Excavation

Upon completion of tank and piping removal activities, CAPE will import backfill from a source that offers native soil that can be certified to be free of anthropogenic impacts. If certification is not available from the source, CAPE will test the imported fill in accordance with the HDOH *Guidance for Soil Stockpile Characterization and Evaluation of Imported and Exported Fill Material* dated October 2017 (HDOH 2017a). CAPE will obtain prior approval of the NAVFAC Hawaii CTO COR/ACOR/DON SMEs for the import backfill source. Prior to placing backfill material, CAPE will isolate native soil from backfill material using plastic sheeting or other suitable material, such as geomembrane. The excavation will be backfilled with gravel and clean fill. Backfill will be direct-loaded into the excavation area and compacted with several passes of heavy equipment. Graded surfaces will be smoothed to prevent ponding/damming of water. The backfill material must be compatible with the standard compaction requirements for vehicle traffic. The site and staging areas will be restored in accordance with JBPHH Environmental requirements and CAPE will replace the galvanized metal fence and gate that is located within the excavation site footprint if removed or otherwise impacted during work.

6.10.2 Round 2 Excavation

Upon completion of Round 2 soil removal activities, CAPE will import backfill from the Grace Pacific LLC Makakilo Quarry. This source has previously been certified to be free of anthropogenic impacts. The existing certification letter and backfill specifications are provided as Appendix B. Backfill will be direct-loaded into the excavation area and compacted with several passes of heavy equipment.

Graded surfaces will be smoothed to prevent ponding/damming of water. The imported backfill material will consist of 3/8-inch to 3/4-inch gravel in the excavation interior and Grace Pacific Quarry's graded select borrow fill in the pavement buffer area; outside the existing paved area, compaction will be completed to an assumed of 90% relative compaction based on the material specifications. Backfill within 2-ft below the pavement subgrade buffer will be compacted to a minimum of 95% relative compaction per ASTM D1557. Backfill compaction testing will be conducted as specified by AGE.

The site and staging areas will be restored to match existing grades and completed with hydromulch spread at less than one inch or similar material in accordance with JBPHH Environmental requirements. CAPE will replace the galvanized metal fence and gate that is located within the excavation site footprint if removed or otherwise impacted during work. CAPE will also replace the removed section of asphalt pavement in accordance with the specifications provided on the SGD/AGE drawings. Pavement will consist of 4-inches of asphalt concrete (or the thickness that matches the existing grade, whichever is greater) underlain by a 2-inch thick aggregate base course.

6.11 Site Surveying

Surface elevations and site contours will be documented with a topographic survey conducted post excavation. The limits of excavation will be staked out in the field for the surveyor to document.

6.12 Demobilization

Once site restoration is complete and graded surfaces are smoothed to prevent ponding/damming of water, all BMPs and construction-related equipment will be removed from the Site in preparation for the final acceptance inspection by the DON. It is anticipated a dry decontamination method will be utilized for construction equipment prior to demobilization; however, the appropriate equipment decontamination method used will be at the discretion of the SSHO/QCO. Further details of decontamination procedures are discussed in the APP. Site cleaning work will include repair of any erosion or run-off-related damage; removal of all materials, such as excess construction material, wood, debris, and other foreign material; and removal of all construction equipment.

Following equipment decontamination, all on-site equipment, material, and personnel will be demobilized from the project Site. The final inspection with NAVFAC Hawaii personnel will be conducted following the completion of the fieldwork. A Closure Report (Section 11) will be prepared by CAPE summarizing all fieldwork activities.

7.0 Waste Management Plan

This WMP addresses the management and disposal requirements for wastes generated during this task order. Wastes will require management and disposal in a manner that is consistent with state and federal law and that minimizes potential hazards to the public. This plan describes methodologies and procedures that will be implemented to handle, manage, and dispose of waste during the project.

7.1 Types of Waste

This section describes the types of waste that will potentially be generated as part of this project.

7.1.1 Green Waste

Vegetation clearing will be performed prior to Round 1 excavation of the concrete tanks and Round 2 soil removal. Prior to vegetation removal in Round 2, a bat and bird survey will occur (see 9.2.4)

Green waste generated during the vegetation clearing will be chipped and disposed of in accordance with JBPHH green waste disposal guidelines and/or Hawaiian Earth Recycling acceptance criteria. The green waste will be loaded onto trucks and removed from site. The green waste will be disposed of off-site at Hawaiian Earth Product's disposal facility on Oahu. If any life stage of the Coconut Rhinoceros Beetle (CRB) is found or suspected, CAPE will stop clearing activities and notify the COR, FEAD, and Hawaii Department of Agriculture Pest Hotline. A CRB identification sheet is provided as Appendix C. In accordance with the March 2022 Joint Base Pearl Harbor-Hickam Green Waste Policy Memorandum, cleared green waste will be transported off-site within 24 hours. If the 24-hour period includes overnight, the material will be staged on-site in a fully enclosed container for immediate next-day delivery to Hawaiian Earth. Vehicles transporting green waste will utilize truck tarps and will be inspected by CAPE personnel prior to leaving the Site. When loading materials for offsite shipment, CAPE's employees will take precautions to prevent green waste spillage. Any spilled debris will be immediately cleaned up from the ground, and green waste will be brushed off the dump trucks before allowing the trucks to leave the Site.

7.1.2 Contaminated Soil

During Round 1, potentially contaminated soil immediately adjacent to the concrete underground tanks and connecting pipe is to be removed from the tank excavation. Soil samples will be collected prior to excavation to determine waste classification, which is expected to be disposed of as non-hazardous waste at PVT Land Company, LTD landfill (PVT Landfill), located in Waianae, Hawaii.

During Round 2 soil excavation activities, contaminated soils and overlying asphalt pavement from the area shown on Figure 6 between approximately 5 and 30 ft bls will be transported to the PVT Landfill for disposal as non-hazardous waste under the existing waste profile for the Site.

7.1.3 Liquid Wastes

During Round 1 activities, liquid wastes removed from the concrete tanks, if present, are expected to potentially contain petroleum hydrocarbons. This waste will be removed using a vacuum truck and containerized in either a truck or 55-gallon drums for off-site disposal by subcontractor PCS.

7.1.4 Tank Debris

During Round 1 activities, debris generated from the dismantling and removal of the concrete tanks and adjoining piping are anticipated to be classified as non-hazardous waste. Tank debris will be immediately loaded into trucks and disposed of off-site to PVT Landfill.

7.1.5 Personal Protective Equipment

Used PPE will be bagged and disposed with non-hazardous debris and trash.

7.1.6 Investigation-Derived Waste

Any non-disposable sampling equipment and excavation tools used during site activities will be decontaminated using techniques that minimize the generation of investigation-derived waste (IDW). Any IDW generated during waste characterization sampling will be disposed with the waste soil or liquids.

7.1.7 Non-Hazardous Debris and Trash

Any site debris and trash generated by site activities will be collected by CAPE and disposed of off-site. Measures will be taken to control the generation of excess waste. Smaller trash bins/barrels with lids will be set up in the support areas for domestic rubbish. Burning of solid waste will not be allowed. Upon completion of the project, the entire site and adjacent work areas will be patrolled for any debris, trash, or litter, and such items will be removed.

Sanitary waste will be collected by a vendor licensed to provide and clean/empty temporary sanitary waste facilities. Sanitary waste from the portable toilets will be collected by a vendor licensed to provide and clean/empty temporary sanitary waste facilities.

7.1.8 Unanticipated Wastes

If unanticipated and potentially hazardous material is encountered, CAPE will assess the unidentified material and, following discussions with the COR, will determine the appropriate analytical methods for identification of hazardous compounds. CAPE will then obtain samples of the material for analysis. If the material is identified as a hazardous waste based on the analytical results, the appropriate treatment and/or disposal will be determined. Sampling, analysis, handling, transportation, and disposal of any hazardous material(s) will be completed in accordance with pertinent federal and local regulatory requirements.

7.1.9 Equipment Fueling and Lubricants

All equipment fueling and maintenance operations will be performed in a manner as to reduce the potential for a release of fuel or lubricants. No fuel tanks will be stored on site. Equipment will not

be “topped off” by the fueling person (operator). Only qualified operators knowledgeable in fueling procedures and equipment fuel tank capacity will fuel equipment. A spill kit will be located on site in the event of a spill. The COR will be immediately notified of any spills.

All lubricant (engine oil, hydraulic oil, transmission oil, etc.) replacement and filling will be performed only by qualified personnel knowledgeable with the particular piece of equipment in the designated area. Used oil pans having capacity of at least 1.5 times the equipment oil reservoir capacity will be used for draining oil. Spill kits will be located at oil replacement/filling areas in obvious locations. The service companies that service the equipment will be responsible for disposing of the used oil.

7.2 Waste Profiling

Waste characterization samples will be collected as described in Section 8.1. After receipt of results, CAPE will prepare the soil profile and waste manifests for impacted soil classified as non-hazardous waste and/or RCRA hazardous waste for review and approval by the NAVFAC Hawaii JBPHH Environmental Compliance and Coordination Branch prior to disposal. For both non-hazardous waste and RCRA hazardous waste, CAPE will coordinate with the NAVFAC Hawaii FEAD to witness the waste loading and inspection activities to the DON’s satisfaction. Upon satisfactory review of the above activities, the NAVFAC Hawaii JBPHH Environmental Compliance and Coordination Branch representative will sign the waste manifests on behalf of the government; however, signature authority may be delegated by the DON to CAPE personnel for non-hazardous waste manifests.

7.3 Transportation

Solid waste will be transported off government property and disposed of in compliance with 40 CFR 260, in addition to state and local requirements for solid waste disposal. A Subtitle D RCRA-permitted landfill is the minimum acceptable off-site solid waste disposal option. Solid waste disposal off site must comply with the most stringent local, state, and federal requirements, including 40 CFR 241, 243, and 258.

Waste designated as non-hazardous for disposal at a Subtitle D facility will be transported to PVT Landfill by the following licensed transporter:

Pineridge Farms, Inc.
855 Umi Street
Honolulu, HI 96819
[redacted]
808-566-8349

Facilities and transportation carriers are subject to acceptance by the EPA and the DON, as well as subject to satisfactory performance. Failure to follow site and regulatory traffic requirements by personnel and waste carriers may result in suspension or ban of the same entities from the project site.

Before relinquishing waste to the transporter identified above, the QCO will review the manifest to verify that it is complete and signed by the DON. The DON may elect to delegate signatory authority to CAPE, in which case the QCO will receive training and a delegation letter from the DON. Verification that the transporter is permitted to transport the type of waste being shipped and that the driver has a current, valid commercial driver's license will be conducted. A copy of the original manifest will be retained and given to the generator, and a photocopy will be retained for the project files.

Before waste leaves the Site, each shipment will be thoroughly inspected to ensure that the packaging, marking, labeling, handling, and placarding of waste complies with federal, state, and local laws and regulations. Vehicles transporting solid waste will be inspected to verify that they have the appropriate placards, that the transporter is permitted to haul the subject waste type, and that each truck tarp (if required) is securely fastened before the truck is allowed to leave the Site. All on-site activities will be supervised by CAPE, and all stages of waste handling by the disposal contractor will be documented. All wastes will be packaged, marked, labeled, and placarded in accordance with federal and state regulations.

All vehicles used for transporting non-hazardous waste/materials will be in good working order, watertight, and have current inspections. The Construction Quality Control Systems Manager (CQCSM) will inspect all the vehicles at the Site prior to loading.

When loading waste materials for offsite shipment, CAPE's employees will take precautions to prevent waste spillage. Excavators or wheel loaders will be used to transfer sediment to dump trucks. Any spilled sediment will be immediately cleaned up from the ground, and clumps of sediment will be brushed off the dump trucks before allowing the trucks to leave the Site.

A 24-hour Emergency Response Contact phone number will be provided on all hazardous material shipping papers in accordance with 49 CFR 172 Subpart G, "Emergency Response Information." The telephone number will be monitored at all times while the hazardous material is in transit (including storage incidental to transportation). The number will be answered by a person who is knowledgeable of the hazardous material and has comprehensive emergency response and incident mitigation information or has immediate access to a person possessing such knowledge and information.

Emergency response information pertaining to the transfer of hazardous materials will be in accordance with 49 CFR 172.600, Subpart G, "Emergency Response Information." Incident reporting and emergency response phone numbers will be verified and provided by CAPE and subcontractors prior to startup of work.

Potential hazards that could occur during hauling include traffic accidents resulting in the spillage of waste. For any hazardous material incidents, the requirements included in 49 CFR 171.15 will be followed. If a reportable incident occurs, CAPE or the person in control of the hazardous material (transporter) will provide a telephone notification to the National Response Center (1-800-424-8802) at the earliest practicable moment, but no later than 12 hours after a reportable incident. Also, the NAVFAC Hawaii FEAD will be immediately notified via e-mail and phone of any incidents or spills.

Measurement for disposal will be by ton of waste transported to the disposal facility. Measurement will be verified with certified weight tickets from the onsite scale at the disposal facility. The weight will be recorded and submitted daily as part of the DQCR and in a Waste Tracking Log.

7.4 Waste Tracking Documentation

After the waste leaves the Site, a clear audit trail will be maintained for the entire disposal operation including, but not limited to, the following:

- Manifest copy(ies);
- Driver information and truck numbers;
- Profile sheet(s); and
- Weight tickets.

A waste tracking log will be maintained and will list all waste materials stored on site and will track off-site disposal. This list includes the description, quantity, waste classification, date that the waste was shipped, disposal facility, method of disposal, and date of disposal/destruction. The log will be maintained in the field office and will be used as a checklist for inspections to ensure all documentation is in place prior to off-site disposal of waste. CAPE will also maintain the NAVFAC waste documentation forms and monthly solid waste disposal report as noted in Specification 01 57 19.

7.5 Record Keeping

Records concerning waste management will be maintained by the DON. CAPE will be responsible for collecting, reproducing, electronic filing and any other record keeping, and data management. RCRA requires documentation of cradle-to-grave management of hazardous waste through a recordkeeping system that tracks shipment of hazardous waste, from the point of generation to final disposal, using a manifest document. Various EPA and DON recordkeeping requirements also apply to waste stream determinations, inspections, manifests, exception reports, Land Disposal Restriction reports, annual hazardous waste reports, and training records. The COR is responsible for maintaining these records in an organized manner and must be able to make them available to the EPA upon request.

7.5.1 Manifests and Exception Reports

Due to the cradle-to-grave liability for generators of hazardous waste disposal, DON policy requires copies of all waste manifests, signed by the generator and the transporter, to be maintained on site for a minimum of three years, and to be archived thereafter for the life of the Site. All Exception Reports, associated correspondence, and other off-site disposal documentation will also be recorded and maintained indefinitely. Original waste manifests will be forwarded to the KO or designated representative.

7.5.2 Waste Stream Documentation

EPA regulations require waste stream documentation forms and profiles, with supporting documentation, to be kept for a minimum of three years.

7.5.3 Training Records

DON policy requires training records to be kept for current employees throughout their employment and for a minimum of five additional years beyond the date of separation. Training records for employees with waste management responsibilities will include the name of the employee; the job title, description, and hazardous waste management duties; a description of the initial and any continuing training requirements for the position; and documentation of satisfactory completion of required training and/or work experience required for the position. Training documentation requirements apply to the following types of training:

- General awareness training;
- 40-hour training for hazardous waste operations and emergency response (HAZWOPER);
- 8-hour HAZWOPER refresher training, required annually;
- Annual Hazardous Waste/RCRA training;
- ECATTS;
- Stormwater Inspector Training; and
- DOT Training.

7.6 Waste Minimization Plan

Waste minimization practices that will be implemented for all stages of the project from waste generation to disposal are described below.

7.6.1 Reducing Consumption of Energy and Natural Resources

Reduction in the consumption of energy and natural resources will be accomplished by not allowing equipment to run idle for extended periods of non-use.

7.6.2 Reducing Waste Generated in the Office

Methods to reduce waste generated in the office include the following: using electronic fillable forms when possible to reduce the amount of paper used; printing directly on envelopes instead of using labels; and reusing single-sided paper. CAPE will purchase recycled products where applicable. All practical efforts will be made to participate in government-sponsored recycling programs.

8.0 Sampling and Analysis Plan

8.1 Waste Characterization Sampling

Prior to Round 1 excavation, one composite waste characterization sample will be collected. The composite sample will be comprised of 20 aliquots. The aliquots will be collected every 4 ft from four 20-ft deep borings (Figure 4). Aliquots will be combined into a clean laboratory provided sample container. Each sample container will be affixed with a sample label provided by the laboratory, which shall include the minimum following information: Sample ID, time, date, and initials of sampler.

A laboratory test report, completed profile, and other supporting documentation will be submitted to the PVT Landfill for review. Profiles will be reviewed and signed by the generator (DON) prior to submittal to PVT Landfill.

The analyses listed in Table 8-1 will be required for disposal. This list is based on previously documented contamination, and PVT Landfill policies and procedures for contaminated soil. The list of waste characterization samples in Table 8-1 was not amended in Round 2. Due to generator knowledge and existing documentation from previous studies/assessments, sampling/testing for additional analytes identified in Specification 01 74 19 was not necessary.

Table 8-1: Soil Waste Characterization Criteria

Lab:	**PACE National, 12065 Lebanon Rd, Mt. Juliet, TN 37122**					
Matrix:	**Waste Characterization, Soil**					
Analyte	**CAS No.**	**Non-Hazardous Criteria**	**Criteria Reference**	**Laboratory Specific Limits**		
				LOQ	**LOD**	**MDL[a]**
TPH, Method 8015B/C/D, Units: mg/kg						
ORO	—	NA	PVT Landfill	0.274	2	4
DRO	—	NA	PVT Landfill	1.61	4	8
GRO	—	NA	PVT Landfill	0.0217	0.55	0.15
TCLP VOCs, Method 1311/8260, Units: mg/L						
Benzene	71-43-2	0.5 mg/L	HAR § 11-261-24	0.000467	0.001	0.002
Ethylbenzene	100-41-4	NA	PVT Landfill	0.000737	0.002	0.004
Toluene	108-88-3	NA	PVT Landfill	0.0013	0.003	0.006
Xylenes, Total	1330-20-7	NA	PVT Landfill	0.00088	0.00325	0.0065
TCLP Metals (RCRA 8)[b] , Method 1311/6010C/7470A, Units: mg/L						
Cadmium	7440-43-9	1.0 mg/L	HAR § 11-261-24	1	0.196	0.049
Chromium	7440-47-3	5.0 mg/L	HAR § 11-261-24	1.3	0.372	0.216
Lead	7439-92-1	5.0 mg/L	HAR § 11-261-24	1.5	0.88	0.222

Notes:

[a] Method Detection Limit (MDL) – For each chemical not detected, an MDL is calculated. The sample-specific MDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the MDL value is reported for non-detected chemicals rather than reporting the QL.

[b] Resource Conservation and Recovery Act (RCRA) establishes allowable toxicity characteristic limits for hazardous waste. HAR § 11-261-24 uses the same toxicity characteristic limits as RCRA.

Definitions:

DRO = Diesel Range Organics
GRO = Gasoline Range Organics
ORO = Oil Range Organics
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit

8.2 Confirmation Sampling

Confirmation samples will be collected from the excavated area once the tanks, connection piping, surrounding soil and additional Round 2 soils are removed. Sampling requirements for confirmation sampling are based on the HEER TGM, specifically Section 3.4 *Selection of Decision Units* (HDOH, 2016), Section 4.2 *Use of Multi-Increment (MI) Soil Samples to Characterize DUs* (HDOH, 2021).

8.2.1 Round 1 Confirmation Sampling

Multi-incremental sampling will be conducted in each tank area once the tanks are removed as well as in the pipeline trench. Two Decision Units (DUs) will be established from each excavated tank area, and two along the excavated connecting pipe, for a total of six DUs. A DU is a targeted area from which samples are to be collected and decisions made based on the resulting data. The approximate locations of each DU are shown in Figure 5. Each MI sample will consist of both floor increments and sidewall increment locations.

A minimum of 30 increments will be collected per MI sample. A systematic random increment collection scheme will be used. Refer to the relevant sections of the Hazard Evaluation and Emergency Response (HEER) Office TGM for detailed guidance on designation of DUs [Section 4, as well as the collection and evaluation of Multi-Increment (MI) soil samples (Section 5)].

A triplicate set of MI samples will be collected in 10 percent of DUs (i.e., from one of the six DUs).

Bulk MI samples collected in the field should be kept to a maximum mass of approximately 2 kilograms. Each 30-increment MI sample will be collected into clean laboratory provided sample container. Each increment should be approximately 50 grams, or approximately 1 US fluid ounce (30 milliliters).

HEER TGM Section 5.6 specifies that the collection of soil samples to be tested for VOCs is similar to that described for nonvolatile contaminants, except that increments are placed in an extraction solution (provided by the laboratory) in the field.

Samples will be shipped on ice packed cooler to the laboratory.

8.2.2 Round 2 Confirmation Sampling

Confirmation sampling of the sidewalls and floor of the larger excavation will be collected using MI techniques as described in Section 8.2.1, except aliquots will be collected from the excavator bucket, which will be used to scrape sample material from the floor or sidewall. The anticipated dimensions of the excavation are a polygon with roughly four sides (north, south, east, and west), as shown in Figure 6.

No sidewalls samples will be collected from the 0-5 ft depth interval. From 5-30 ft bls, each sidewall will be divided into halves or quadrants based on size. The floor will be divided into halves. A total of 14 DUs are anticipated. Refer to Figure 6 for the location and depth of each DU. Samples from the DUs in the 5-12 ft depth range will be collected from the sloped sidewalls.

Triplicate MI samples will be collected from two DUs (one floor DU and one sidewall DU). Due to the excavation depth, sampling personnel will not enter the excavation area and samples will be collected directly from the excavator bucket. Sample handling and shipping requirements are summarized in Section 8.2.4.

8.2.3 Confirmation Sample Handling

Confirmation samples will be collected in the containers summarized in Table 8-2 below and stored in iced coolers pending shipment to the laboratory. Due to Department of Transportation/International Air Transport Association shipping requirements to the US mainland, a maximum of 18 TPH/VOC jars containing methanol (i.e., 3 samples) can be packed in one cooler. A copy of the signed COC for each cooler will be taped inside and the cooler will be sealed with packing tape and laboratory-supplied custody seals for overnight shipping via FedEx.

Although overnight shipping will be requested, it is frequently not feasible for FedEx to accommodate overnight shipping requests. The field team will assume a minimum of two days in transit, and coolers must be packed with sufficient ice and shipped several days in advance of hold time expirations.

Table 8-2: Confirmation Sample Handling Requirements

Lab: PACE National, 12065 Lebanon Rd, Mt. Juliet, TN 37122
Matrix: Soil

Analytical Group	Analytical Method	Containers Per Sample	Total Sample Volume	Preservation Requirements (per container)	Anticipated Number of Samples[a]	Maximum Holding Time[b]
TPH (DRO, ORO) and PAHs (Naphthalene, 1-Methylnaphthalene, 2-Methylnaphthalene)	8015B/C And 8270C SIM	one double bagged 1-gallon Ziploc bag	1,500 g	Ice (4° C)	30	14 days
TPH (GRO) and VOCs (BTEX only)	8015D	six 4-ounce glass jars with Teflon lined lids	150 g (25 g per jar)	25 mL Methanol and Ice (4° C)	30	14 days

Notes:
[a] Including triplicates
[b] From the time of sample collection to preparation or extraction

Definitions:
TPH= total petroleum hydrocarbons
BTEX = benzene, ethylbenzene, toluene, and xylenes
DRO = diesel range organics
ORO = oil range organics
GRO = gasoline range organics
PAHs = polycyclic aromatic hydrocarbons

8.2.4 Confirmation Sample Analysis

Soil chemical analysis will be performed by Pace Analytical, a DoD National Environmental Laboratory Accreditation Program (NELAP) certified laboratory, located in Mount Juliet, Tennessee. The Mount Juliet laboratory will manage the project, compile all deliverables and invoices, and perform all analyses (including MI sample preparation). The laboratory NELAP certification is provided as Appendix D.

Once received by the laboratory, MI samples will be processed within a 5-day turnaround time and in accordance with Pace Analytical procedure ENV-SOP-MTJL-0112 (Appendix E) which is summarized below:

1. Dry the samples
    a. Lay the entire contents on a clean pan.
    b. Spread the samples in a thin layer. Using gloved hands to break up any clumps.
    c. Place the tray in a drying hood or well-ventilated area at room temperature.
    d. When the samples appear dry enough that they can be sieved without caking, reweigh the sample.
    e. Continue reweighing the sample every 12 hours; once two consecutive weights within 10-percent difference are achieved the sample is considered dry.
2. Sieve the samples
    a. Break up samples with gloved hands if needed. Re-record the weight.
    b. Sieve the entire dried sample through the appropriately sized sieve. Remove large rocks, vegetation and twigs.

3. Collect incremental samples

    a. Place the entire sample onto pan to a 1-centimeter thickness.

    b. Using a subsampling tool, take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material.

Following laboratory preparation, samples will be analyzed for the parameters included in Table 8-3.

Table 8-3: Soil Confirmation and Backfill Criteria

Lab: PACE National, 12065 Lebanon Rd, Mt. Juliet, TN 37122
Matrix: Soil

Analyte	CAS No.	Project Action Limit (PAL): HDOH Leaching and Groundwater Protection Action Level [a] (mg/kg)	HDOH Final EAL [a] (mg/kg)	Laboratory Specific Limits		
				LOQ	LOD	MDL[b]
TPH, Method 8015B/C/D, Units: mg/kg						
DRO (middle distillates)	—	940	220	1.61	4.0	8.0
ORO (residual fuels)	—	1,000	500	0.274	2.0	4.0
GRO (gasolines)	—	700	100	0.0217	0.055	0.15
VOCs, Method 8260 VOCs, Units: mg/kg						
Benzene	71-43-2	0.3	0.3	0.000467	0.001	0.002
Ethylbenzene	100-41-4	0.9	0.9	0.000737	0.002	0.004
Toluene	108-88-3	0.78	0.78	0.0013	0.003	0.006
Xylenes, Total	1330-20-7	1.4	1.4	0.00088	0.00325	0.0065
PAHs, Method 8270SIM, Units: mg/kg						
Naphthalene	91-20-3	3.1	3.1	0.00408	0.01	0.02
1-Methylnaphthalene	90-12-0	0.89	0.89	0.00449	0.01	0.02
2-Methylnaphthalene	91-57-6	1.9	1.9	0.00427	0.01	0.02

Notes:

[a] PALs are based on DOH Environmental Action Levels for Groundwater Protection of a Drinking Water Resource, Table A-2, Soil Action Levels (Potentially impacted groundwater is a current or potential drinking water source; surface water body is located within 150 meters of the release site) (HDOH, 2017b).

[b] Method Detection Limit (MDL) - For each chemical not detected, an MDL is calculated. The sample-specific MDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the MDL value is reported for non-detected chemicals rather than reporting the QL.

8.2.5 Confirmation Sample Reporting

The closure report will include the following substantive elements from the HDOH Guidance (HDOH, 2017b):

- Summary of sampling methodology and analytical results from confirmation sampling, including:
    1. Identification of DUs (e.g., horizontal and vertical dimensions)
    2. Number and location of sampling increments in DUs

3. Number and location of replicate samples
4. Summary of laboratory analytical results and copy of laboratory data reports
5. Chain of custody documentation
6. Any additional information that may be necessary to assess chemical levels in the confirmation samples;

- Evaluation of sample data with respect to EALs; and
- Identity/signature by party responsible for evaluation of each confirmation sample.

8.3 Quality Assurance/Quality Control and Data Validation

The CAPE Chemistry Services Lead will review field documentation and COCs to verify that sampling procedures are conducted in compliance with this SAP. Environmental Data Services (EDS) will conduct data validation activities for excavation confirmation samples. EDS will prepare Stage 4 data packages for confirmation samples for upload to the Naval Installation Restoration Information Solution in Naval Electronic Data Deliverable format. QA/QC procedures for laboratory analysis will be conducted in accordance with the current version of the DoD Quality Systems Manual (QSM) (DoD, 2019), the USEPA analytical method, and applicable laboratory generated limits.

CAPE will initiate corrective action procedures if conditions are identified that may impact data quality. Corrective action may be initiated by the Chemistry Services Lead or PM to address violations of field procedures documented in this SAP and violations of established laboratory procedures or controls. If significant problems are identified by the laboratory or the project team that affect the usability of the data, the CAPE Chemistry Services Lead and the PM will notify the COR within 24 hours or the next business day. For laboratory QC variances, the laboratory will notify the CAPE Chemistry Services Lead within 24 hours of identified variance(s). If quality control variances contradict the minimum requirements of the DoD QSM, then the CAPE QCM and PM will contact the COR via telephone and/or e-mail to discuss and receive approval for the variances within 7 days of notice of variance. All corrective actions will be approved by the appropriate project personnel, and documentation will be maintained with the project files.

9.0 Environmental Conditions/Environmental Protection Plan

9.1 General Overview and Purpose

The purpose of this ECEPP is to ensure that the terrestrial and aquatic environments are protected during the removal of the concrete tanks. The ECEPP presents site background information, outlines duties and lines of communication for site personnel, and specifies procedures and plans. The ECEPP has been prepared in accordance with Performance Work Statement (PWS) Section 2.3.3.

9.2 Communications

CAPE will ensure that everyone working on the project site, including CAPE employees and subcontractors, is aware of the project activities and potential hazards, and has received adequate training in safety, health, and environmental procedures. Prior to performing any work on site, all employees and subcontractors will receive a mandatory briefing that covers S&H and environmental requirements. In addition, these topics will be covered during each preparatory phase inspection before work commences. Additional meetings will be conducted for new personnel and when site conditions change. All employees and subcontractors will be required to report to CAPE's SSHO for S&H issues, and to CAPE's QCO for environmental issues.

9.2.1 Protection of Natural Resources

South Halawa Stream is located to the north and downslope of the excavation area. CAPE understands our responsibility to preserve the natural resources within the project boundaries and adjoining areas. As practical, the Site will be restored to an equivalent or improved condition upon completion of work.

9.2.2 Land Resources

To the extent practical, land surfaces outside the work area limits will not be disturbed without approval from the CTO COR and other DON representatives (e.g., ACORs, other subject matter experts). Equipment lay-down or work pads will be as small as practical without jeopardizing worker safety or productivity.

9.2.3 Cultural Resources

No cultural artifacts are expected to be discovered during the demolition, excavation, and removal of the concrete tanks and surrounding soil. In the unlikely event that such material is encountered, ground-disturbing activities in the immediate area of the discovery will be halted, and the DON will be contacted for further instructions.

9.2.4 Wildlife Resources

Removal activities will be managed to minimize interference with, disturbance to, and damage of wildlife resources. CAPE will do everything possible to ensure that field activities do not interfere with any wildlife or wildlife habitats. For Round 2, clearance of trees greater than 15 ft tall will not occur until an infrared survey for the Hawaiian hoary bat (*Aeorestes semotus*) has been completed.

Immediately prior to (i.e., the morning of) vegetation clearance, CAPE will conduct a bat and bird survey (starting at approximately 0430 and lasting until approximately 0730 or unless otherwise agreed to or directed by the United States Fish and Wildlife Service (USFWS)). The bat survey will include infrared survey clearance by a qualified wildlife biologist and arborist for Hawaiian hoary bats (*Aeorestes semotus*), which are protected under the Endangered Species Act) prior to trimming or removal of trees greater than 15 ft tall within an approximately 25-ft buffer of the work footprint (where applicable, disturbed footprint and Halawa stream limits exist). If any observations of nesting or potential bat roosts are documented, CAPE will coordinate with the Navy on next steps which could include proceeding with avoidance and minimization measures as established or agreed to with the USFWS, notifications to the USFWS via the DON, or similar. No trimming/removal will occur where bats are documented. If bats are documented, prior to trimming/removal, CAPE will resurvey to confirm if the bat is still present. If the bat is still present, work will not commence until documentation that the bat has left, or the breeding season is complete.

In addition, CAPE will comply with the requirements of the Migratory Bird Treaty Act and Executive Order 13186, *Responsibilities of Federal Agencies to Protect Migratory Birds*. CAPE will verify that trees and bushes being removed do not contain the active nests of migratory birds. If a nest is encountered during vegetation clearance, CAPE personnel will not disturb the birds or their nest, and the CAPE PM will immediately report the sighting to the DON for further instructions. In coordination with the DON, next steps which could include establishing nest buffers or having onsite monitors. Nesting season for migratory birds takes place from March through August, peaking in May and June. For Round 1, vegetation clearance will be minimal. For Round 2, vegetation clearance is greater, but work is occurring near the end of the breeding season. CAPE does not anticipate encountering any migratory birds during this project.

9.3 Air Quality Monitoring Plan

No federal or state air permit is required for the on-site consolidation and sampling activities being conducted for this project.

The concrete tanks and connection piping along with surrounding soil removal will be performed in a manner that reduces the potential for fugitive emissions and complies with the substantive requirements of these regulations. Mitigating measures will be undertaken to reduce air emissions, control potential particulate emissions, and prevent air quality impacts during these activities. An example of one measure will be to use a water truck to spray down soil during excavation activities for dust control.

The principal source of short-term air quality impacts will be earthmoving operations associated with the removal. These impacts will be minor and of short duration. Fugitive dust may be generated during these activities. BMPs will be implemented to contain fugitive dust.

9.3.1 Pollution-Generating Equipment

The following pollution-generating equipment is expected to be used during the execution of this task order.

- 1 excavator;
- 1 loader;
- 1 Skidloader;
- 1 2,000-gallon water truck; and
- 84-inch Roller.

9.3.2 Air Pollution Engineering Processes

The only planned air pollution-generating processes involved in this project are the routine operations of the pollution-generating equipment listed in Section 9.3.1 above. No spray painting, abrasive blasting, or demolition will be conducted. Dust control is discussed in Section 9.3.4 below. Hours of operation of equipment and quantities of materials will be tracked as described in the PQCP (Section 10). Emissions will be limited by avoiding idling equipment unnecessarily. Work will be planned to keep idling to a minimum, and equipment that does not need to be idling will be shut down.

9.3.3 Monitoring

The risk of contaminant emissions posing a threat to public health is low due to the remote location of the concrete tanks. Perimeter air monitoring is not planned. See the SSHP for protocols for identification of perimeter or personnel air monitoring standards and planning.

9.3.4 Dust Control

Dust production will be reduced or eliminated by engineering controls first, and PPE will be used by personnel, if necessary, for nuisance dust issues.

During the removal of the concrete tanks and surrounding soil, BMPs will be implemented to ensure sediment and dust are controlled. When trucks are loaded with excavated materials, precautions will be taken to ensure that spillage does not occur by minimizing drop heights and using spotters. Additionally, the operators will be supervised and will not be allowed to overfill the excavator bucket. Field personnel will visually inspect trucks prior to departing the Site to ensure that excess or loose material is not tracked onto access roads within the Red Hill Bulk Fuel Storage Facility.

The primary activity that will generate airborne dust is expected to be the hauling and handling of material during excavation. Dust will be primarily controlled using water spray application with a water truck. Dust suppression will be provided during all truck loading activities by wetting the soil and spraying any visible dust with a water mist. An off-site water source will be used to refill the water truck for dust control.

9.3.5 Odor Control

If present, odors from construction activities will be controlled at all times with dust control measures (water spray). The SSHO will monitor odors and may stop work if odors are excessive or conditions (such as high wind) cause odors to spread. If deemed necessary by the SSHO, CTO PM, or ACOR, other methods may be implemented to limit odors on the Site, but that is not anticipated at this time.

9.4 Noise Control Plan (Sound Intrusion)

CAPE will identify work areas where hearing protection is required (if any) and post signs and provide hearing protection to workers as necessary. All internal combustion engine equipment utilized on this project will be fitted with standard noise-reducing mufflers. If noise resulting from the consolidation and berm construction activities exceeds an average of 85 decibels over 8 hours of duration (dBA), then the 85 dBA line will be identified, and personnel will post hearing and hazard warning signs where applicable. Noise will also be limited by avoiding idling equipment unnecessarily. Work will be planned to keep idling to a minimum, and equipment that does not need to be idling will be shut down. If complaints are received, the SSHO will notify the CTO PM, COR, ACOR, and CAPE PM; track the complaints; and work to provide a solution if practicable.

Site personnel working in the immediate area of operating equipment are required to use hearing protection (e.g., foam ear plugs) whenever noise exposures exceed 85 dBA. Noise levels decrease dramatically over distance and are not anticipated to pose a risk to bystanders located outside of the limits of disturbance. Refer to the SSHP for information regarding noise monitoring and hearing protection requirements.

9.5 Stormwater Pollution Prevention Plan

No NPDES permit is required for this project. However, this Stormwater Pollution Prevention Plan describes the stormwater management and control measures that CAPE will implement to prevent the discharge of any pollutants to the stormwater system or ocean as required by the EPA's Construction General Permit (EPA, 2022).

9.5.1 Stormwater Management and Control

Wattles (temporary berms) will prevent migration of soils and pollutants from moving outside the site boundaries as shown in Figure 3. Berm locations may be field-adjusted and additional erosion and sediment control practices (i.e., silt/fence, hay bales, sidewall sloping, dust control, etc.) will be performed as necessary. CAPE may construct and/or install additional erosion and sediment control BMPs to divert stormwater from exposed areas and prevent migration of soil and pollutants from moving outside the boundary limits of the Site.

During field activities, the CAPE EM will inspect disturbed areas and sediment controls every 7 calendar days, and within 24 hours (during normal site work hours) of a 24-hour total rainfall accumulation of 0.25 inches or more based on a nearby representative weather station. The inspection will ensure the controls are in place, are functioning, and are adequate. If sediment

inspections indicate that a control is not functioning properly, the control will be replaced or modified promptly.

9.5.1.1 Soils Prone to Erosion

CAPE will institute measures to prevent the runoff of soils prone to erosion at the Site. Once the necessary controls have been installed, the CAPE QCO will walk the project site with the DON to ensure that the DON is satisfied with the location and amount of protection that is installed. CAPE will discuss with the DON what measures are expected to be in place and will maintain these features throughout the project.

9.5.1.2 Mechanical Retardation and Control Runoff

CAPE will be prepared to protect storm drains and/or culverts by placing silt fencing around any storm drain inlets located within work areas, to ensure sediment does not wash into and clog the drains. No storm drains have been identified near the immediate Site area. The nearest drainage ditch is located approximately 60 ft west of the excavation area.

9.5.1.3 Other Best Management Practices for Stormwater Management and Control

Site personnel will be trained on the importance of stormwater management control and the methods to be employed; and the training will be documented in the applicable preparatory phase inspection(s).

Miscellaneous rubbish and debris generated during site activities will be marshaled on a daily basis. This housekeeping activity will prevent miscellaneous rubbish and pollutants from becoming entrained in stormwater effluent as a release to the environment.

All equipment fueling and maintenance operations will be performed in a manner as to reduce the potential for a release of fuel or lubricants. All equipment will receive fuel from an aboveground storage tank (AST) filled by a qualified fuel-service company. Equipment will not be “topped off” by the fueling person (operator). Only qualified operators knowledgeable in fueling procedures and equipment fuel tank capacity will fuel equipment. Fire extinguishers and spill kits will be located at the fueling tank, in an obvious location. The COR, CTO PM, ACOR, and CAPE PM will be immediately notified of any spills. Additionally, the AST will meet the following guidelines and requirements:

- EPA’s Spill, Prevention, Control, and Countermeasure (SPCC) requirements (40 CFR Part 112);
- A secondary containment will be installed (i.e., berms, dikes, liners, double-walled tank, catchment basin);
- Adequate safeguards against fire, overfills, and damage will be installed;
- State, local fire codes, and federal regulations will be met;
- Daily inspections will be performed, and an applicable material safety data sheet will be kept at the Site;
- There will be no smoking/open flames in fueling areas; and
- Equipment will not be operated within 10 ft of the AST.

Equipment will also be fueled once it has been cooled to ambient temperature with the motor off. Contingency measures will be provided for potential spills and discharge from handling potentially hazardous materials on site. Vehicle refueling will only occur outside the EZ and will be executed with proper grounding and spill prevention techniques, as discussed in the next section.

9.5.2 Prevention of Releases to the Environment

CAPE will provide contingency measures for potential spills and discharge from handling potentially hazardous materials on site. The only hazardous materials anticipated are diesel fuel and lubricants for heavy equipment and small tools. These chemicals will not be stored on site overnight. Equipment refueling and lubrication activity will only occur outside the EZ and will be executed with proper grounding and spill prevention techniques. Fire extinguishers and a spill kit will be posted in close proximity to any fueling activity; and a spill pan, bucket, etc. will be placed below each equipment's fueling port. All heavy equipment will be inspected daily for leaks, loose hydraulic hoses, equipment requiring repair or replacement, functionality of emergency instruments and switches, etc. Additionally, if equipment is parked on site overnight, equipment will have oil pans placed underneath. In the event of a release of a hazardous substance, the following will be implemented:

- Notify the COR, CTO PM, FEAD CM/ET, and ACOR immediately;
- Notify the Regional Dispatch Center (RDC) at (808) 474-1271. If the spill can be cleaned up without assistance from the Navy Region Hawaii (NAVREGHI), the caller shall so inform the dispatcher at the RDC when making the call so that emergency response personnel are not dispatched. If assistance is needed, the RDC shall send the appropriate personnel and equipment. Depending on the nature of the spill, emergency response may involve the DON On-Scene Coordinator;
- Notify the National Response Center at 1-800-424-8802 within 24 hours of the incident if the spill is greater than the reportable quantity according to 40 CFR 302;
- Notify the local Fire Department (911) immediately of the incident;
- Provide methods, means, and facilities to prevent contamination of soil, water, air, structures, equipment, or material from any release due to the Contractor's operations the same day of the incident;
- Provide equipment and personnel to perform emergency measures to mitigate spills and control their spreading immediately after the incident;
- Dispose of contaminated materials within 10 work days; and
- Provide a decontamination program to clean previously uncontaminated areas within 15 work days.

All spills on NAVREGHI property shall be reported to the RDC by the responsible party causing the spill. The RDC will contact the personnel listed on the oil and hazardous substance spill checklist. If any spill involves a reportable quantity, CAPE will complete the required reports and notifications.

9.5.2.1 Equipment Required

CAPE will have the following equipment on site at all times in order to handle hazardous material releases:

- Noncombustible absorbent, such as vermiculite or floor-dry;

- U.S. Department of Transportation (DOT)-approved containers for temporary storage of spilled or leaking materials;
- Shovels, brooms and other hand tools; and
- PPE (poly-coated Tyvek®, nitrile gloves, goggles, caution tape, sheeting, etc.).

9.5.2.2 Contingency Plan

The following actions/measures will be implemented during a spill response action:

- Notify the DON immediately;
- Take immediate measures to control and contain the spill using above-mentioned equipment and materials;
- Isolate and contain hazardous spill areas;
- Deny entry to unauthorized personnel;
- Do not allow anyone to touch spilled material;
- Stay upwind;
- Keep out of low areas;
- Keep combustibles away from the spilled material;
- Use water spray to reduce dust, as needed;
- Perform cleanup activities as directed by the DON using competent/qualified personnel;
- Take samples for analysis to determine that cleanup is adequate;
- If released from tanks, prevent discharge beyond site boundaries; and
- Any other actions as needed.

9.5.2.3 Notification of Spills and Discharges

CAPE will make all spill notifications under federal and local regulation, including, but not limited to 40 CFR 110, 302, 355, 370, 372, etc., immediately upon discovery of a spill or discharge. CAPE will notify the appropriate authorities, unless notified that the DON will take responsibility for notification. A report, submitted to the DON no later than 24 hours after a release, will include the following items:

- Description of material spilled, including identity, quantity, and a copy of the waste disposal manifest;
- Exact time and location of the spill, and the description of the area involved;
- Containment procedures utilized;
- Description of the cleanup procedures employed at the Site, including disposal of spill residue;
- Summary of CAPE's communications with other agencies; and
- Determination if the spill has been reported to the DON and/or reportable, and the date that the report to the appropriate agency was made, and the name of the agency representative who accepted the report.

If any spills exceed the reportable quantity for a particular material, the DON will be required to provide notification within 24 hours to the State Emergency Response Commission [(808) 586-4444] and the Local Emergency Planning Committee [(808) 523-4121]. Follow-up written notification would be required within 30 days.

The QC procedures that CAPE personnel will follow during the performance of this task order are detailed in the PQCP located in Section 10.0. The primary function of QC management is to ensure tasks are performed in accordance with the PWS and within the defined schedule and budget. This is achieved through the execution of a realistic plan to ensure that the required standards of quality will be met.

CAPE will implement the requirements of the PQCP during all phases of the project. This includes inspections, tests, and periodic QC meetings at the Site to track and report progress. The PQCP also outlines procedures to help monitor contract compliance during the project utilizing three phases of control.

10.0 Project Quality Control Plan

This PQCP addresses the requirements for the performance of work under Contract N62742-16-D-1807, CTO N6274222F0135, *Concrete Tank Removal Project at Red Hill Fuel Storage Facility, Joint Base Pearl Harbor-Hickam, Hawaii*" for the NAVFAC Hawaii. It reflects company commitment to quality and is the blueprint for ensuring quality deliverables for this CTO. It identifies requirements and assigns responsibilities for ensuring that the project objectives are met. To do this, it describes or references the controls, quality procedures and processes, and guidelines to be followed.

This PQCP has been developed in accordance with the PWS and specifications sections 01 57 19, 01 74 19, and 02 81 00 for this CTO, and the Program Quality Control Plan.

10.1 Quality Control Program Organization

CAPE has assembled a proven team to execute this project. CAPE will perform all site project management, QC management, S&H management, logistics, property control, and cost and schedule controls in-house and provide technical and management services locally as required. All subcontractor personnel will adhere to the requirements of this plan through their respective quality organizations and will ultimately report to the site-specific QCO concerning QC issues.

An organizational chart showing the reporting relationship of personnel involved in this project is provided in Figure 7. The QCO is responsible for the QC organization as supported by the Program QA Manager and additional QC staff as dictated by CTO directives. The QC team and organization will include the Program QA Manager, Program Manager (PGM), Program S&H Manager, and PM. On-site construction QC organizations will include the QCO and SS. The responsibilities, authorities, and reporting requirements of these positions are identified in the following. The PGM and Program QA Manager will determine selection of qualified and experienced personnel for project-level positions.

To ensure independence from operations, the QCO will report directly to the Program QA Manager. This structure ensures that quality issues within the project are not compromised by project cost/schedule considerations and are allowed to immediately rise to a higher program and corporate level of responsibility in the organization for resolution, as necessary.

When necessary, CAPE will revise the PQCP to reflect changes and submit the changes to the COR for acceptance.

10.2 Meetings

While performing work under this CTO, CAPE will take part in various progress meetings to update the DON, and when applicable, other stakeholders, on the progress, results, deviations or modifications, and recommendations for the fulfillment of the scope of work.

10.2.1 Coordination and Mutual Understanding Meeting

Prior to start of the site work, the QCO will meet with the CTO COR or designated representative to discuss the PQCP. The objective of this meeting will be to develop a mutual understanding of the system details, including the forms for recording the QC operations, administration of the system for both on-site and off-site work, management, and the interrelationship of CAPE's management and control with the government's QA, production, and the SS's duties with the KO or designated representative. As a minimum, project personnel required to attend will include the PM, SS, and QCO. This meeting will likely be held in conjunction with the Pre-Construction Meeting.

10.2.2 Quality Control Meetings

The QCO will conduct regular QC meetings with the CTO COR, PM, and SS after the start of fieldwork and until the CTO fieldwork is complete. The meetings will be recorded in the DQCR. These meetings may be held in conjunction with other meetings (i.e., tailgate safety meetings, progress meetings). At a minimum, the following shall be covered at each meeting:

- Minutes from the previous QC meeting;
- Work or testing accomplished since the last QC meeting;
- Rework items identified and/or completed since the last QC meeting;
- Current schedule, work to be accomplished before the next QC meeting, and the documentation required;
- Review the status of submittals;
- Coordination and/or notifications required for proposed work;
- Completion dates for rework items;
- Preparatory, initial, follow-up phase inspections, and testing required;
- Resolve any QC/production issues with required documentation; and
- Revisions to the PQCP, such as changes in procedures.

10.2.3 On-site Meetings

During the course of fieldwork, meetings may take place, whether planned or unplanned, at the Site to discuss specific issues or concerns. The QCO will be responsible for documenting discussions and decisions made at the meeting on the DQCR.

10.2.4 Meeting Minutes

Minutes of all meetings will be prepared describing the general discussion, decisions, changes in approach, and action items. CAPE will submit the meeting minutes to participants within the timeline specified by the PWS. As a general rule, minutes will be distributed within 48 hours of the meeting.

10.3 Control

The following are the Definable Features of Work (DFWs) identified for this CTO:

- Mobilization and Site Preparation;
- Site Clearing;
- Waste Characterization (Round 1 only);
- Tank Removal, and Disposal (Round 1 only);
- Soil Excavation and Disposal (Round 2 only);
- Confirmation Sampling; and
- Site Restoration and Demobilization.

10.3.1 Three-Phase Control System

The three-phases of control are the core of the Construction Quality Management System and are the means by which CAPE ensures construction, including that of subcontractors and suppliers, complies with the requirements of the contract. The three-phase control system includes the Preparatory Phase, the Initial Phase, and a Follow-Up Phase and are explained below. Three-phase control system will be conducted by the QCO for each DFW of the construction work as per the specifications. A DFW is a task that is separate and distinct from all other tasks and has a specific set of control requirements (e.g., concrete placement, soil compaction, and instrumentation).

Each control phase provides an opportunity to prevent deficiencies that result in nonconformance. Implementation of the three-phased QC process is the responsibility of the project team. For each task assignment, specific charts, checklists, etc., will be prepared to assist the QCO in ensuring that the work elements are properly performed.

10.3.2 Preparatory Phase

The preparatory meeting will be held before the start of construction and is typically conducted by the QCO (or designated member of the QC team) with other applicable site personnel in attendance. The NAVFAC Hawaii CTO COR will also be invited to attend the meeting. Any subcontractors involved in the DFW will participate in this review as well. The QCO will send out notice in advance of the meeting so that appropriate personnel may attend.

During this phase, it is imperative to review the specifications and drawings to ensure all preparatory steps have been taken, to verify that submittals have been prepared and reviewed, qualified manpower is assigned, testing controls are prepared and in place, and safety issues have been identified and addressed. Specifically, this phase will include:

- Review of each paragraph of applicable specifications and drawings;
- Review of provisions that have been made to provide required control testing and inspection;
- Concurrence required project training and qualifications are complete (QC, S&H, technical, etc.);
- Review to ensure that all materials and/or equipment have been tested, submitted, and approved;
- Review of applicable permit status and requirements;

- Examination of the work area to ensure required preliminary work has been completed and is in compliance with the contract;
- Examination of all required materials, equipment, and sample work to ensure they are on hand, conform to approved shop drawings or submitted data, and are properly stored;
- Review of the appropriate AHA to ensure safety requirements are met;
- Discussion of construction method and establish construction tolerances and workmanship standards;
- Check to ensure the portion of the plan for the work to be performed has been accepted by the client; and
- Discussion of the initial control phase.

10.3.2.1 Initial Phase

This phase of inspection will document the completeness and acceptability of the particular items at the beginning of the work activity after a representative portion of the items has been completed. This inspection is typically held with most of the personnel present during the preparatory meeting. This is the time to ensure that CAPE and subcontractor personnel/workers understand, through immediate inspection, the contract standards and the standards of workmanship desired. If there is a difference of opinion in the interpretation of contract requirements, this is the time to settle the issue. The initial inspection phase is a practical method of performing preventive inspection and resolving conflicts. The following will be accomplished during this phase:

- Review of minutes of the preparatory meeting for open items;
- Check of the work to ensure that it is in full compliance with the project requirements;
- Verification of adequacy of controls;
- Verification of required control inspection and testing are being performed;
- Establishment of level of workmanship and verification that it meets the desired acceptable workmanship standards. Comparison with required sample panels as appropriate;
- Resolution of all differences between personnel and subcontractors;
- Check of safety to include compliance with and upgrading (if necessary) of the SSHP and AHA;
- Review applicable AHAs with each worker;
- Preparation of documentation of the initial phase inspection, including a narrative description of detailed inspection procedures, minutes of meetings, inspection results, corrective measures, etc., using forms presented in Appendix F; and
- The initial phase will be repeated for each new crew to work onsite or any time acceptable specified quality standards are not being met.

10.3.2.2 Follow-up Phase

Follow-up inspection and testing is geared toward a level of effort to verify the continuation of project compliance and standards of workmanship established during the previous two phases. This inspection is typically conducted periodically by the QCO (or designee) to ensure a continuation of satisfactory quality standards. Follow-up inspections will be made a matter of record in the DQCR and inspection checklists for each DFW. The QCO will closely monitor the actual

field testing, verifying proper procedure technique, sample handling, and Chain of Custody (COC), if required. The QCO will monitor testing results and compare them with the project requirements, and if acceptable, document the results and provide a timely authorization to proceed with subsequent work. If the QCO determines the test results or work is otherwise not acceptable, they will notify the PM for stopping work and initiating a conformance action. Final follow-up checks will be conducted, and all deficiencies will be corrected before the start of additional DFWs.

10.3.2.3 Additional Preparatory and Initial Phases

Additional preparatory and initial phases will be conducted on the same DFW if the quality of ongoing work is unacceptable; if there are changes in the applicable QC staff, on-site supervision, or work crew; if work on a DFW is resumed after a substantial period of inactivity; or if other problems develop.

10.3.3 Control for Off-Site Work

When necessary to perform the scope of work for a project, work may take place off site. This may include analytical testing at a certified laboratory, data validation, manufacturing of material or equipment for use or installation at a project site, work performed by subcontractors in fulfilling the project objectives, or other activities. In such cases, CAPE will control the quality and workmanship of products or equipment developed offsite by implementing the same control measures as work that takes place at a project site. This will ensure products and materials meet the overall quality and technical standards for the CTO as well as the contract and ensure materials delivered to the job site conform to expected standards.

When a piece of equipment or material is produced offsite, the CAPE Project Engineer and/or QCO will oversee development of the design plans for such equipment or materials. He/she will review shop drawings, calculations, or other relevant data produced by the manufacturer or specialty subcontractor, may visit the manufacturer during or after production to ensure proper construction and adherence to acceptable tolerances, and document changes, modifications, or corrections to the design or construction.

10.3.4 Deficiency Management

The CAPE quality program evaluates the effectiveness of the company's QC program and ensures continuous improvement in the quality of CAPE's work. The primary goals of CAPE's quality program are to prevent non-conformances and to facilitate continual process improvement. If the first goal is not achieved, the identified deficiencies or non-conformances will be corrected in a timely and cost-effective manner in order to prevent their recurrence. The PQCP will include provisions for preventing quality issues, facilitating process improvements, as well as identifying, documenting, and tracking deficiencies until corrective actions have been verified.

10.3.4.1 Preventive Measures

While the entire QC program is directed toward problem prevention, certain elements of the program have greater potential to be pro-active. The primary tools for preventing problems include:

- Employee qualification and training;
- Preparatory, initial, and follow-up inspections; and
- Equipment calibration and maintenance.

10.3.4.2 Continual Improvement

All project personnel are encouraged to recommend improvements to work processes and techniques. The intent is to identify activities that are compliant but may be performed in a more efficient or cost-effective manner. Typical quality improvement recommendations include identifying an existing practice that can be improved (e.g., a bottleneck in production) and/or recommending an alternative practice that provides a benefit without compromising the standards of quality.

Project personnel may bring their recommendations to the attention of project management or QC staff through verbal or written means; however, deviations from established protocols will not be implemented without prior written approval by the PM and concurrence of the QCO. When a staff-initiated recommendation results in a tangible benefit to the project, the PM will give public acknowledgement. All variances from the contract specifications or drawings will be documented using a Field Change Request (FCR) form (Appendix F). This form will identify the variance, proposed change, technical justification, and cost and schedule impacts (to be determined by the PM as applicable). FCR forms may include either additions or deletions of work elements and may be identified by CAPE, NAVFAC Hawaii, or its agent. All FCR forms will be submitted to the COR to be reviewed, approved, and signed prior to implementation.

10.4 Testing and Inspection

10.4.1 Inspection

CAPE will conduct QC inspections as a part of the Three-Phase Control Process to verify whether quality-related activities comply with this QC Plan. Internal inspections will address activities performed by CAPE. External inspections will address activities performed by project subcontractors, equipment and material suppliers.

The inspection program is established to provide the following:

- An objective and independent evaluation of compliance with established policies and procedures (WP, AHAs, APP, etc.); and
- A mechanism for verifying the implementation of corrective actions recommended as the result of inspections.

Personnel performing QC inspections will be knowledgeable about and have received training in QC techniques, methods, this QC Plan, applicable regulations, and will be technically knowledgeable related to the process being inspected. Inspections will be performed in accordance with written procedures or checklists. Personnel performing QC inspections will not have direct responsibilities in the areas they are assessing.

System and performance inspections will be undertaken. System inspections will evaluate the components of the QC system including evaluating items such as approach and adequacy of the

preparation step, inspection of the schedules and plan delivery dates, and tracking systems for QC activities. Performance inspections evaluate actual QC activities such as design control, on-site data gathering, instrument calibration and control, inspection and testing activities, and documentation.

Inspecting QC personnel will document inspection results, which will be reviewed by the PM. When unsatisfactory or nonconforming conditions or items are found, the responsible organization will implement corrective actions in a timely manner. Previously unsatisfactory areas will be re-inspected to ensure that satisfactory corrective actions have been completed. The results of the inspections will be shared with the team with regard to needed rework and lessons learned.

Records of all inspections, re-inspections, and corrective actions will be maintained and controlled as QC records.

10.4.1.1 Receiving Inspection

Incoming materials will not be used until they have been inspected or tested and found to be conforming to specified requirements, have not been damaged during the delivery process, and storage provided is adequate to preserve the material. Inspection will be provided by a member of the QC Team.

10.4.1.2 Punch-Out Inspection

A "punch list" of items that do not conform to the approved drawings and specifications will be prepared and included in the QC documentation. The list of deficiencies will include the estimated date by which the deficiencies will be corrected. The QCO or staff will make a second inspection to ascertain that all deficiencies have been corrected. Once this is accomplished, the QCO will notify the government that the Facility is ready for the government pre-final inspection.

10.4.1.3 Pre-Final Inspection

The government and QCO will perform the pre-final inspection to verify that the work is complete and the Site is ready to be closed. A government pre-final punch list may be developed as a result of this inspection. The QCO will ensure that all items on this list have been corrected before notifying the government so that a final inspection with the client can be scheduled. Any items on the pre-final inspections will be corrected in a timely manner. These inspections and any deficiency corrections required by this paragraph will be accomplished within the time slated for completion of the entire work or any particular increment of the work if the project is divided into increments by separate completion dates.

10.4.1.4 Final Acceptance Inspection

The CAPE PM will notify the COR when all deficiencies of the punch list from the pre-final inspection have been corrected. The COR will be notified of the scheduled date at least 14 calendar days before the final acceptance inspection and will be given CAPE's assurance that all specific items previously identified are acceptable, along with all remaining work under the contract. The CAPE SS, QCO and the COR will be in attendance at the final acceptance inspection. Additional

government personnel including, but not limited to, those from base/civil facility engineer user group and major commands may also be in attendance.

10.4.2 Instrumentation/Equipment Testing, Inspection, and Maintenance

All equipment used at a project site shall be manufacturer-calibrated to national standards. At the start of each work day, calibration shall be verified and tested for functionality. Equipment found to be damaged, inoperable, or out of calibration shall not be used until the discrepancy is corrected and verified by the QCO. Once equipment has been used, it shall be maintained following manufacturer's recommendations.

10.5 Quality Control Certification

10.5.1 Contractor Quality Control Report Certification

Each DQCR will contain the following statement signed by the QCO:

"On behalf of the Contractor, I certify that this report is complete and correct, and equipment and material used and work performed during this reporting period is in compliance with the contract drawings and specifications to the best of my knowledge except as noted in this report."

10.5.2 Invoice Certification

CAPE will provide with each payment request to the COR or designated representative a certification, attesting that the work for which payment is requested is compliant with contract requirements.

10.5.3 Completion Certification

Upon completion of the work under a CTO, the PM will present a certificate of completion stating that the "*work has been completed, inspected, tested, and is in compliance with the contract*."

10.6 Quality Control Documentation

The QCO serves a critical role in documenting, reporting, and storing data in support of the QC process and its effectiveness in attaining a high-quality work product.

10.6.1 Reporting Requirements

The QCO will complete and maintain the QC records to provide factual evidence of compliance with project requirements and to document all QC activities, including maintaining a record of all tests and inspections performed. These records include all work performed by subcontractors and suppliers. The QCO will sign each report and provide copies to the PM and NAVFAC Hawaii COR. The primary QC reports are listed in the sections below.

10.6.1.1 Daily Contractor Production Report

The SS is responsible for preparing and submitting a Daily Contractor Production Report documenting the activities and progress to the COR the next working day after each calendar day

that work was performed. The report will document work performed and results used to gauge progress and adherence to schedule and cost. The Daily Contractor Production Report will be signed and dated by the SS and document, at a minimum, the following:

- Date of report, report number, name of contractor, contract number, title and location of contract and task order, and superintendent present;
- Weather conditions in the morning and afternoon, including maximum and minimum temperatures;
- Personnel (including CAPE, subcontractors, and visitors), materials, and equipment on the work site that day;
- Job safety actions taken and safety inspections conducted (indicate that safety requirements have been met), including the results on the following:
  - Was a job safety meeting conducted? If so, attach a copy of the job safety meeting minutes
  - Were there any lost time accidents? If so, attach a copy of the completed OSHA report
  - Was trenching, scaffold, high-voltage electrical, or high work done? If so, attach a statement or checklist showing inspection performed
  - Was hazardous material or waste released into the environment? If so, attach description of incident and proposed action);
- Equipment and material received and incorporated into the job;
- Construction and plant equipment on the work site, with number of hours used, idle, and down for repair;
- Work performed by CAPE and subcontractor personnel, including volumes, rates, and results;
- Waste or material accumulated or transported offsite on a daily basis;
- Workforce job hours and cumulative hours; and
- Remarks regarding pertinent information, including directions received, problems encountered during activities, work progress and delays, conflicts or errors in the specifications, field changes, safety hazards encountered, instructions given and corrective actions taken, delays encountered, and a record of visitors to the work site.

10.6.1.2 Daily Contractor Quality Control Report

The QCO will prepare a DQCR and deliver to the COR the next working day after each calendar day that work was performed.

The DQCR will provide an overview of QC activities performed each day, including those performed on subcontractor and supplier activities. The DQCR will present an accurate and complete picture of QC activities, document both conforming and deficient conditions, and should be precise, factual, legible, and objective. Copies of the supporting documentation, such as checklists and surveillance reports, COC records, and manifests, will be attached. The report will include the following information, at a minimum:

- Control phase and the DFW;
- Results of preparatory, initial, and follow-up phase inspections;
- Location of DFW and names of personnel present;

- Findings and measurements made to document adherence to project plans and specifications;
- Steps taken to correct non-conformances;
- Results of the three phases of control for off-site work, if applicable, including actions taken;
- Description of daily site work;
- Weather conditions, including ambient temperatures, precipitation, and wind estimates;
- Type and location of tests performed and results of the tests;
- Verbal instructions received from the CTO COR;
- Submittal action;
- Inspection results of delivered equipment and materials;
- List rework items identified but not corrected by close of business;
- Review of subcontractor data, calculations, or drawings;
- Results of meetings or communication with vendors, NAVFAC Hawaii, regulators, or others related to the performance and quality of work;
- Off-site surveillance of fabricated items;
- Remarks containing pertinent information, including directions received, QC problem areas, deviations from the PQCP, construction deficiencies encountered, QC meetings held, acknowledgment that as-built drawings have been updated, corrective direction given by the QCO, and corrective action taken by CAPE; and
- Signature and certification of QCO.

10.6.1.3 Rework Items List

Items requiring rework will be completed in accordance with the Rework Items List. The list will identify the reason or cause for the deficiency and prescribe the appropriate remedy. Once the item has been satisfactorily reworked, it will be closed-out of the Rework Items List and noted in the DQCR. The Rework Items List will be attached to the last DQCR of the month. It will show:

- Items to be reworked;
- Date originally discovered; and
- Date resolved.

10.6.1.4 Monthly Summary Report of Field Tests

The QCO will submit one monthly summary of field tests attached to the DQCR at the end of each month.

10.6.1.5 Quality Control Meeting Minutes

The QCO will submit one copy of the meeting minutes within 2 working days of the meeting.

10.6.1.6 Safety and Health Deficiency Tracking

The SSHO shall establish and maintain a safety and occupational health deficiency tracking system in accordance with 01.A.12.d of EM 385-1-1, *Safety and Health Requirements Manual* (USACE, 2014).

10.6.1.7 Field Logbook

The QCO will be assigned a QC logbook for documenting details of field activities during QC monitoring activities. The logbook will be a bound manuscript with pre-printed page numbers. The QCO will document his daily duties, summarize field activities, including arrival and departure time, note QC tests and results, depict the site layout, and mark locations of tests and other data. The information in the QC logbook will serve as a detailed description of events to aid in the preparation of the DQCR and in addressing follow-up questions that may arise.

All entries made in the QC logbook will be made in ink. Any changes will be made by a single strike through the error and initials. At the beginning of each day, the QCO will begin on the following page in the logbook. Descriptions will be made in coherent English and in a manner that would allow others to recreate the daily events in the absence of the QCO. At the end of each day, the QCO will mark the remainder of the page with a single line and sign across or under it.

10.6.2 Records Management

Records are considered completed documents, validated data, and other materials that provide objective evidence of the quality of items or activities. A document that contains objective information can become a record once it is complete and identified as a record. Records include, but are not limited to:

- Work plans, technical proposals, and other work assignment planning documents;
- Field plans and procedures;
- Training records;
- CTO reports, including letter reports;
- Field logbooks and project notebooks;
- Three phase inspection records;
- Daily QC and Production Reports;
- COC records;
- Audit, surveillance, and independent project self-assessment reports;
- Field change notices; and
- Laboratory data.

Records will be maintained at the project site in a manner that prevents deterioration and provides for the safeguarding of the records. A record indexing system that allows for easy retrieval and provides sufficient information to permit the correlation of records with the items or activities to which they apply will be used. Inactive records will be stored for the mutually agreed upon time after which they are either transferred to NAVFAC Hawaii or disposed of.

Disposition of records is controlled and documented. Records are destroyed only after the proper notification to NAVFAC Hawaii and the approval of the PGM.

10.6.3 Change and Control Procedures

A formal process identifies, documents, and tracks the status of procedural and condition changes in remedial action work. Changes required due to changes in field conditions that differ from those presented in contract documents will be documented by an FCR form.

Proposed changes that have not physically occurred are also documented on an FCR. In instances where the physical work has been completed, the FCR is used to provide the as-built information and allow the PM and their team the opportunity to review the impact of those changes on other components of the work. The FCR should not be confused with a Nonconformance Report (NCR), as outlined in Section 10.8.

The FCR is prepared by the QCO, as required, and routed to the PM for review. The PM will discuss potential changes with the appropriate NAVFAC Hawaii Representative and other client personnel, as required. Before routing, the PM will use the document control system to assign an FCR number, retains a copy for the FCR log and contract files, and then forwards a copy of the FCR to the COR. The QCO will monitor the documentation and provide support.

10.7 Submittals

A Submittal Register will be created and maintained for this CTO. All material submittals will be transmitted in accordance with the instructions pertaining to NAVFAC Hawaii.

Preparation of submittals is the responsibility of the CAPE PM using project staff resources. For preparation of technical submittals, appropriate staff will be chosen who possess the background and knowledge required for ensuring that the technical submittal is complete and accurate. The PGM or PM should require a separate technical peer review for highly technical submittals.

The QCO will review submittals for acceptance with QC requirements before transmittal to either NAVFAC Hawaii or other required approval authorities. Submittals requiring modifications or changes will be returned to the originator, subcontractor, or vendor for correction and resubmission. The QC Manager will approve or disapprove the submittal, as appropriate, and will indicate his action by his signature and date before sending back to originator or forwarding to government for approval.

Submittals from subcontractors and vendors will be reviewed for technical content and accepted as a part of this submittal preparation procedure.

10.7.1 Submittal Register

Submittals will be listed and tracked using the Submittal Register. Submittals include deliverables, whether generated onsite or offsite by CAPE or subcontractor personnel. The Submittal Register will be updated as needed along the project life. The QCO will review the list and ensure its completeness and may expand general category listings to show individual entries for each item. The Submittal Register will be used as the scheduling document and to control submittals throughout the project.

10.7.2 Submittal Schedule

The QCO will monitor a project submittal schedule that reflects the status of project submittals. Submittal activities will be incorporated into the project schedule so that the submittal progress can be tracked in conjunction with the overall progress. During periodic QC meetings or progress meetings, the PM will present an updated schedule, showing both actual field progress and deliverable and submittal schedules.

10.7.3 Submittal Procedure

All submittals will be submitted in format dictated by the PWS or as required by NAVFAC Hawaii. Manufacturer's descriptive data that have more than one model, size, or type or that show optional equipment will be marked to show the model, size, or type, and all optional equipment proposed for approval. Submittals on component items forming a system or that are interrelated will be submitted at one time as a single submittal to demonstrate that the items have been properly coordinated and will function as a unit.

A Material Approval Submittal Form will be completed for submitting shop drawings, equipment data, manufacturer's literature and certifications, and samples of material. Items to be approved will be clearly tabbed or identified. Submittals will be numbered consecutively, by contract, in the space entitled "Submission No." The number, in addition to the Contract No., will be used to identify each Material Submittal. Re-submissions will be indicated in the appropriate block.

Submittals requiring government approval will be identified on the Material Approval Submittal Form and will be stamped and dated by the government when approved and returned to the CTO PM. The QCO will indicate the action by the government on the Submittal Register.

10.7.4 Re-submittal Procedure

The government may require CAPE to re-submit an item found not to comply with the project requirements. When a submittal has been disapproved and/or returned with comments, a subsequent submittal (re-submittal) will be made in the same manner as the original submittal.

10.7.5 Deviations

For submittals that include proposed deviations, the variation will be noted on the transmittal form. CAPE will set forth in writing the reason for any deviations and annotate such deviations on the submittal. The government reserves the right to rescind inadvertent approval of submittals containing unnoted deviations.

10.7.6 Control of Submittals

CAPE will carefully control its procurement operations so that materials and equipment are not ordered until the government has approved the submittal covering the subject material or equipment. Submittal preparation will be planned to allow review and approval time so that materials and equipment are ordered in time not to affect the project schedule.

10.7.7 Internal Review

To emphasize the importance of an independent technical review (peer review) before transmitting a submittal to NAVFAC Hawaii, a QC Review will be implemented by the PM. The PM and the QCO will identify the reviews required for each document to ensure that the checks are performed.

The QCO will monitor the internal review. If differences are found between the author and the reviewer that remain unresolved, the PM will mediate to find a common ground.

10.7.8 Certification

Prior to delivery or use, project submittals will be reviewed and approved by the QCO. The certification and signature of the submittal reviewer will be required prior to QCO approval. All submittals prepared by CAPE or its subcontractors will be reviewed for completeness and compliance with the specifications of the contract. Non-conforming submittals will be returned to the originator for corrective action and re-submittal to the QCO.

10.7.9 Transmittal to the DON

CAPE will transmit submittals via electronic mail and will be accompanied with a highlighted copy of the submittal register form. Transmittal is intended for submitting both "DON approval" and "information only" submittals. Care will be exercised to ensure the proper listing of the contract specification paragraph pertinent to the data submitted for each item.

CAPE will prepare and include a transmittal form with each submittal. The transmittal form will identify CAPE as the contractor, indicate the date of the submittal, and include information prescribed and required by the transmittal form. CAPE will identify submittals with the following information permanently adhered to or noted on each separate component of each submittal and noted on the transmittal form. CAPE will mark each copy of submittals with the following:

- Project title and location;
- Contract number and CTO;
- Section and part number of the section by which the submittal is required;
- Submittal description number of each component of the submittal;
- If a re-submission, an alphabetic suffix on the submittal description to indicate the re-submission;
- Name, address, and telephone number of the subcontractor, supplier, manufacturer, and any other second tier contractor associated with the submittal; and
- Product identification and location in project.

10.7.10 Comment Response Matrix

Upon receiving comments from DON, regulatory, or other outside reviewers, CAPE will prepare a comment response matrix to document each reviewer's comment along with a response describing how the comment will be resolved, incorporated, or addressed. In cases where agreement of the response is not reached easily between CAPE and the reviewer, CAPE may request the COR/ACOR to set up a conference call or on-site meeting with reviewers to discuss their comments. The completed comment response matrix will be submitted to the COR/ACOR and reviewers via electronic mail before revising the actual report, and adequate time will be allocated to permit reviewers to read and accept or revise CAPE responses. At such a time when all responses are accepted by the DON, CAPE will incorporate changes into the document and prepare for re-submission.

10.8 Nonconformance Reporting

10.8.1 Nonconformance Definition

The definition of a nonconforming condition can range from a departure from established requirements to identification of deficient material, assembly, or construction method. All items determined to be nonconforming must be corrected through systematic corrective actions. Any time a condition exists not in compliance with drawings, specifications, codes, workmanship standards, facility requirements, or NAVFAC Hawaii requirements, the nonconformity must be documented, corrected, and closed-out through the following means.

10.8.2 Deficiency Identification

Usually, NAVFAC CTOs require the presence of a QCO onsite who traditionally manages the issue of nonconformance and approves the close-out of corrective actions. If a QCO has not been assigned to the project; the PM will be responsible for managing the nonconformance process. Regardless, all nonconforming items must be reported to the Program QA Manager. Anyone finding a deficient item is responsible for reporting it to the appropriate management staff.

10.8.3 Nonconformance Determination

During routine site activities, the majority of corrective actions can be implemented immediately (within 48 hours) by the work assignment staff and documented in the field logbook. If the condition is not quickly corrected, the individual initiates an NCR as shown in Appendix F and submits it to the QCO or Program QA Manager. The QCO or Program QA Manager will notify the PM and identify the person responsible for implementing corrective action (often the SS), sets a date on which the response is due, and distributes the NCR.

10.8.4 Planning and Implementing the Corrective Action

The responsible person to initiate corrective actions should identify the cause of the problem, if known or suspected, on the NCR. The responsible person should develop a Corrective Action Plan, identify the date the corrective action has been or will be accomplished, describe the action taken on the form, and return the form to the QCO or Program QA Manager by the response due date. If possible, objective evidence that the corrective action has been completed should be included with the NCR response. If this is not possible, the responsible person should return the NCR by the due date and provide the evidence as soon as possible.

10.8.5 Accepting Corrective Action

The QCO or Program QA Manager will review the NCR response to determine the adequacy of the corrective action. If the stated corrective action is unacceptable, the NCR will be returned to the responsible person for further discussion and corrective action.

10.8.6 Verifying Corrective Action

If the evidence provided to the QCO or Program QA Manager concerning completed corrective action is acceptable, the NCR will be signed and dated. If evidence is obtained through an audit,

surveillance, or follow-up review, the individual conducting the follow-up will sign and date the form once the corrective action has been verified.

10.8.7 NCR Distribution

A distribution list for NCRs will be determined at the initial project-planning meeting. At a minimum, distribution will include the COR/ACOR, SS, QCO, PGM, Program QA Manager, PM, and any other applicable client-related individuals.

10.9 Audits and Assessments

Assessments are a learning process intended to increase the user's understanding of the program or system being assessed and to provide a basis for improving such programs or systems. The purpose of assessments is to improve the quality of work by comparing the system or element to the specified requirements. Assessments are conducted at all levels: corporate, contract, project, and activity. Response refers to the actions taken by the assessed organization as a result of the assessment. Typically, responses involve corrective actions to rectify the deficiencies identified in the assessment.

Assignments will be monitored and assessed as deemed appropriate by the Program QA Manager. The following sections identify and describe many assessment types. The requirement to conduct applicable assessment types may be further defined in the work plans and field plans. The PGM, PM, or the Program QA Manager may specify additional assessments as necessary to ensure that the quality of work meets DON and CAPE's expectations. Assessment reports generated under the SBRAC contract will be made available to the NAVFAC COR/ACOR upon request.

10.9.1 Management System Reviews

Management systems reviews are self-assessments conducted annually, or as determined appropriate by the Program QA Manager, to establish whether the quality management structure, policies, and procedures are adequate to ensure quality data. Management systems reviews may cover multiple contracts and quality plans. The primary focus of the management systems review is performance improvement through:

- Fostering individual ownership of the quality program by increasing employee involvement in quality;
- Encouraging employees to routinely identify opportunities for quality improvement;
- Meeting with management, technical, and QA staff to solicit specific suggestions to improve quality, such as more practical implementation methods, procedural modifications, etc.;
- Training the management, technical, and QC staff on quality plan requirements;
- Communicating lessons learned from other management systems reviews; and
- Checking on implementation and effectiveness of the quality program within the office.

10.9.2 Project Audits

Project audits may be conducted on CAPE and subcontractor work activities by QC staff independent of the work activities. The implementation and use of appropriate quality measures

identified in this PQCP, work plans, field plans, specifications, procedures, and any other applicable document specifying requirements can be used for checking during the audit. The auditor will also ensure that obsolete documentation has been removed from project work areas and check briefly on preparation of required deliverables and the condition of project files.

Project auditors will be trained in auditing procedures and authorized by the Program QA Manager. The responsibilities and procedures for planning, conducting, reporting, and closing out audits are specified in Auditing Procedure.

10.9.3 Project Self-Assessments

Project self-assessments are evaluations of work activities conducted by project personnel who are knowledgeable in the project requirements to determine if the technical requirements are being met. They are intended to provide rapid feedback to the project staff to facilitate timely corrective action.

The PGM makes the selection of assignments or activities for project self-assessment and the personnel to conduct them with the assistance of the Program QA Manager or QCO. Project self-assessments are conducted using a standardized or customized checklist.

11.0 Closure Report

A Closure Report will be prepared following the completion of the project. The report will document the removal of the concrete holding tank, concrete leach tank, connection piping, and additional grossly-contaminated soil at the Site. It will include waste characterization and confirmation sampling results, figures, tables, description of any deviations from the WP and a conclusion and decision-making section with a comparison of the confirmation DU analytical results to the project action limits discussed in Section 8. In addition, the report will present appendices with copies of any permits, field work documentation (logs), meeting minutes, laboratory reports, data validation reports, project photographs, and waste manifests. The Closure Report will include the rationale and data used to arrive at conclusions and recommendations and the Quality Assurance (QA)/QC procedures used to check assumptions and verify findings.

12.0 References

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Figures

Figure 1
Site Location
Concrete Underground Tank
Removal at Red Hill Bulk Storage Facility
Work Plan
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

Map Projection:
NAD 1983 StatePlane
Hawaii 3

N

0 700 1,400 2,800

Feet

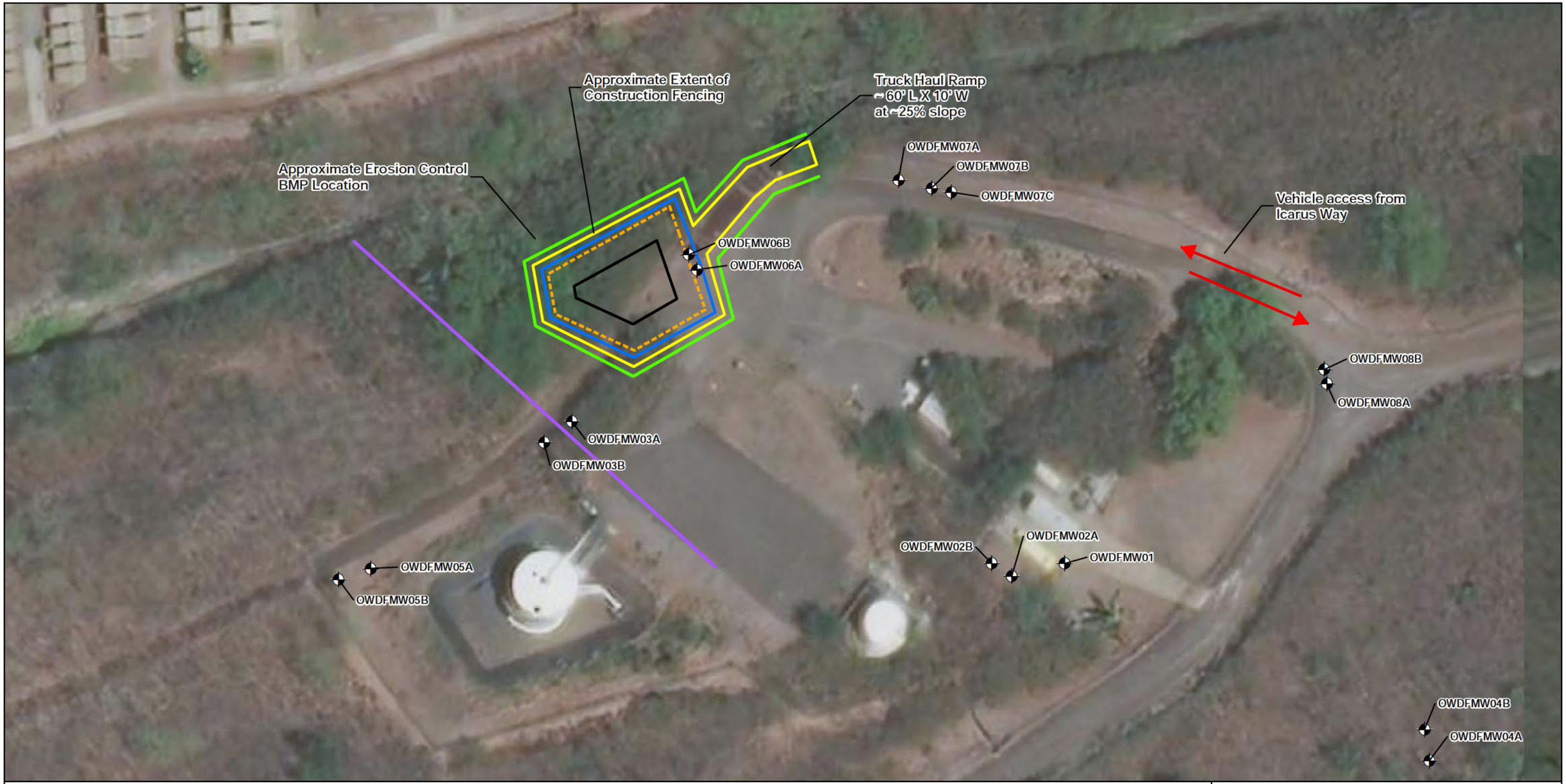
Figure 2
Haul Route
Concrete Underground Tank
Removal at Red Hill Bulk Storage Facility
Work Plan
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

Map Projection:
NAD 1983 StatePlane
Hawaii 3

N

0 175 350 700

Feet



Legend

- OWDF Monitoring Well (approximate location)
- Granular Activated Carbon Lines
- Approximate Erosion Control BMP Location
- Approximate Work Area Extent
- Approximate Extent of Construction Fencing
- Excavation Boundary
- 1:1 Slope Layback 0-12 ft bls

Notes:
Total Excavation Volume = 1,900 Cubic Yards
L x W - length by width
BMP - Best Management Practice

Vegetation clearing will be conducted as needed and chipped green waste transported to the Kalaeloa Bio-Solid Treatment Facility. Pavement and fencing within the work area will be removed and replaced to match existing conditions during site restoration.

BMP and construction fencing/barricade locations will be modified as necessary based on field conditions.

N

0 50 Feet

Round 2
Site Staging Plan

Red Hill Bulk Storage Facility
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

PREPARED BY: [redacted]	CONTRACT NO: N62742-16-D-1807	August 2022
REVIEWED BY: [redacted]	TASK ORDER: N6274222F0135	FIGURE: 3

CAPE

Legend

- Waste Characterization Sample Locations
- OWDF Monitoring Well (approximate location)
- Underground Pipeline
- Underground Pipeline to be Removed
- Holding Tank
- Leach Pit
- Proposed Extent of Excavation
- Drainage Ditch

N

0 25 Feet

Waste Characterization Sampling Locations

Red Hill Bulk Storage Facility
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

PREPARED BY: [redacted]	CONTRACT NO: N62742-16-D-1807	August 2022
REVIEWED BY:	TASK ORDER: N6274222F0135	FIGURE: 4

CAPE

Legend

- OWDF Monitoring Well (approximate location)
- Approximate Location of Granular Activated Carbon Lines
- Underground Pipeline
- Underground Pipeline to be Removed
- Holding Tank
- Leach Pit
- Sampling Decision Units and Extent of Excavation
- Drainage Ditch

N

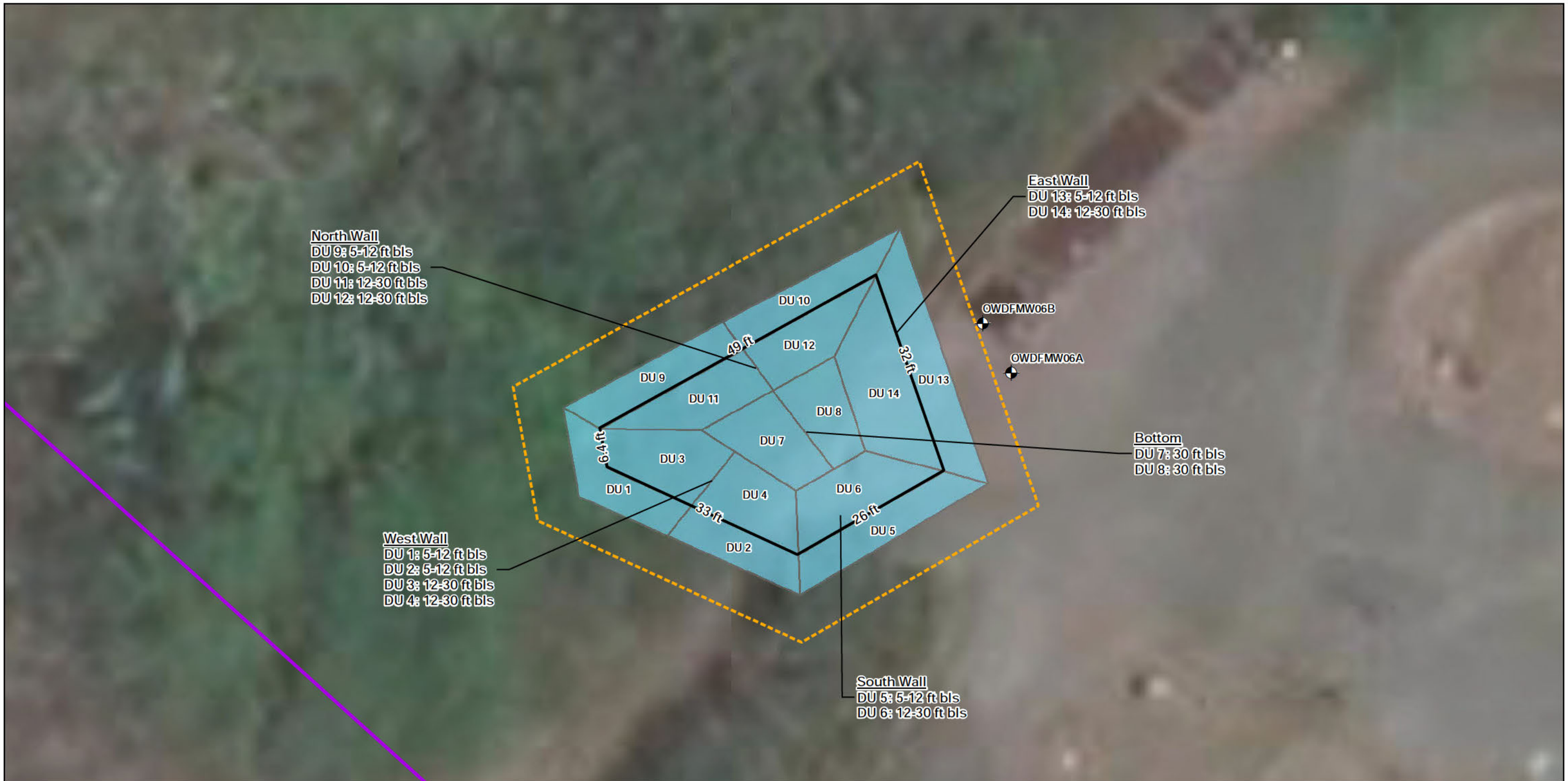
0 25 Feet

Round 1 Confirmation Sampling Decision Units

Red Hill Bulk Storage Facility
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

PREPARED BY: [redacted]	CONTRACT NO: N62742-16-D-1807	August 2022
REVIEWED BY:	TASK ORDER: N6274222F0135	FIGURE: 5

CAPE



Legend

- OWDF Monitoring Well (approximate location)
- Approximate Location of Granular Activated Carbon Lines
- Excavation Boundary
- DU Boundary
- 1:1 Slope Layback 0-12 ft bls

Notes:
Total Excavation Volume = 1,952 Cubic Yards
ft .bls - feet below land surface
DU - Decision Unit
DU dimensions may be field adjusted based on
excavation production rates and sample holding times.

N

0 15 Feet

Round 2 Confirmation Sampling Decision Units

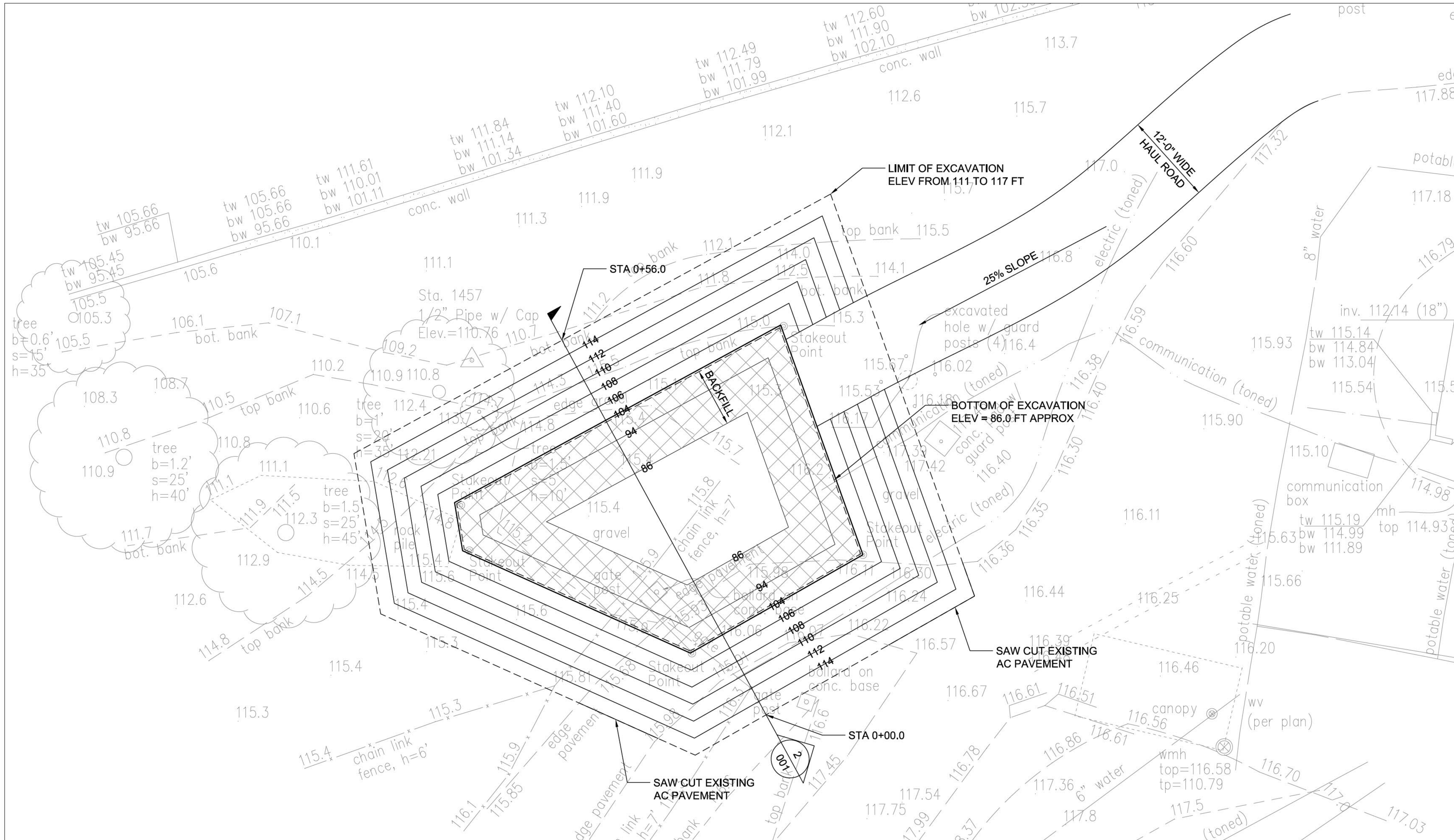
Red Hill Bulk Storage Facility
Joint Base Pearl Harbor-Hickham
Oahu, Hawaii

PREPARED BY: [redacted]	CONTRACT NO: N62742-16-D-1807	August 2022
REVIEWED BY:	TASK ORDER: N6274222F0135	FIGURE: 6

CAPE

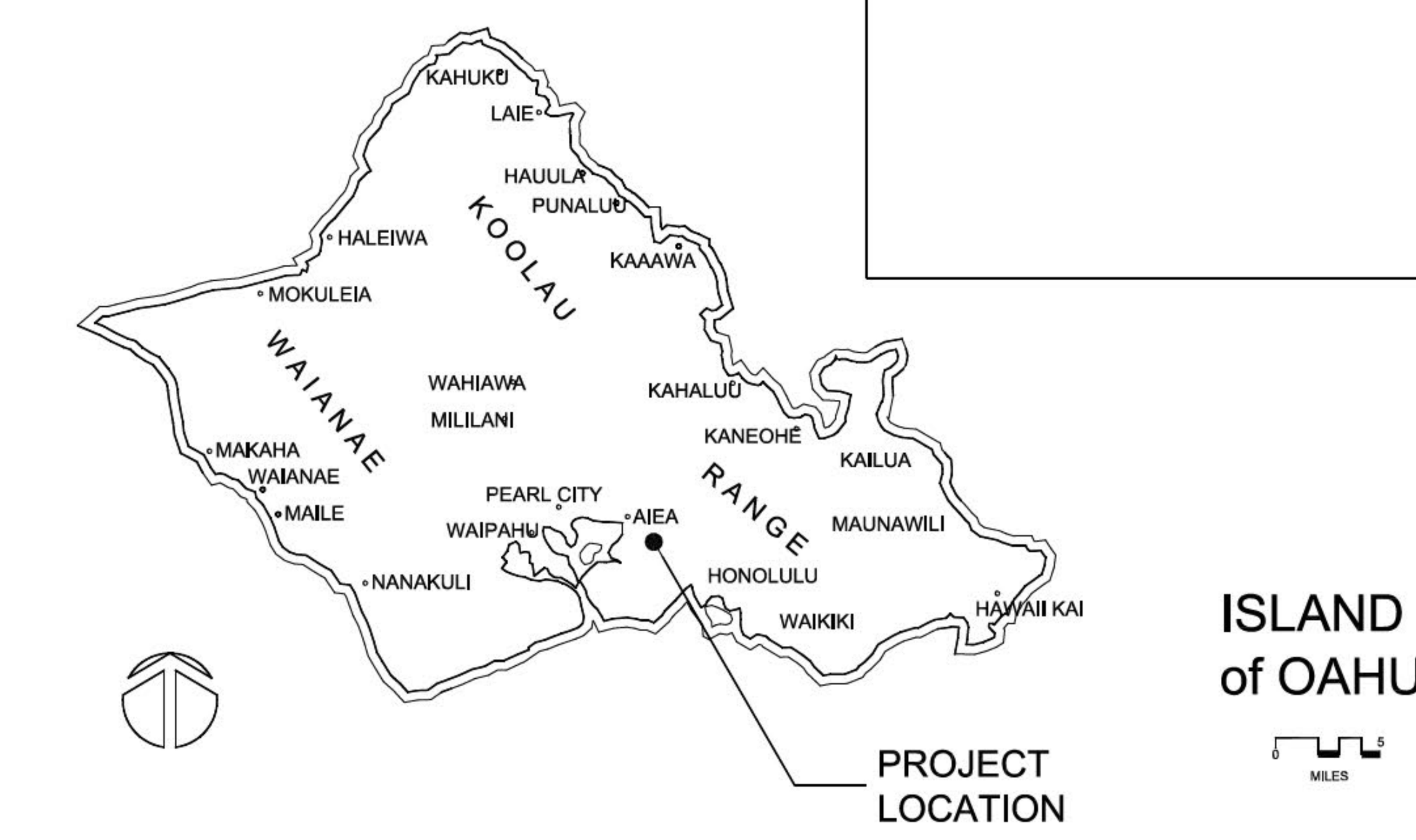
Appendices

Appendix A: Red Hill Excavation Drawings (prepared by AGE)

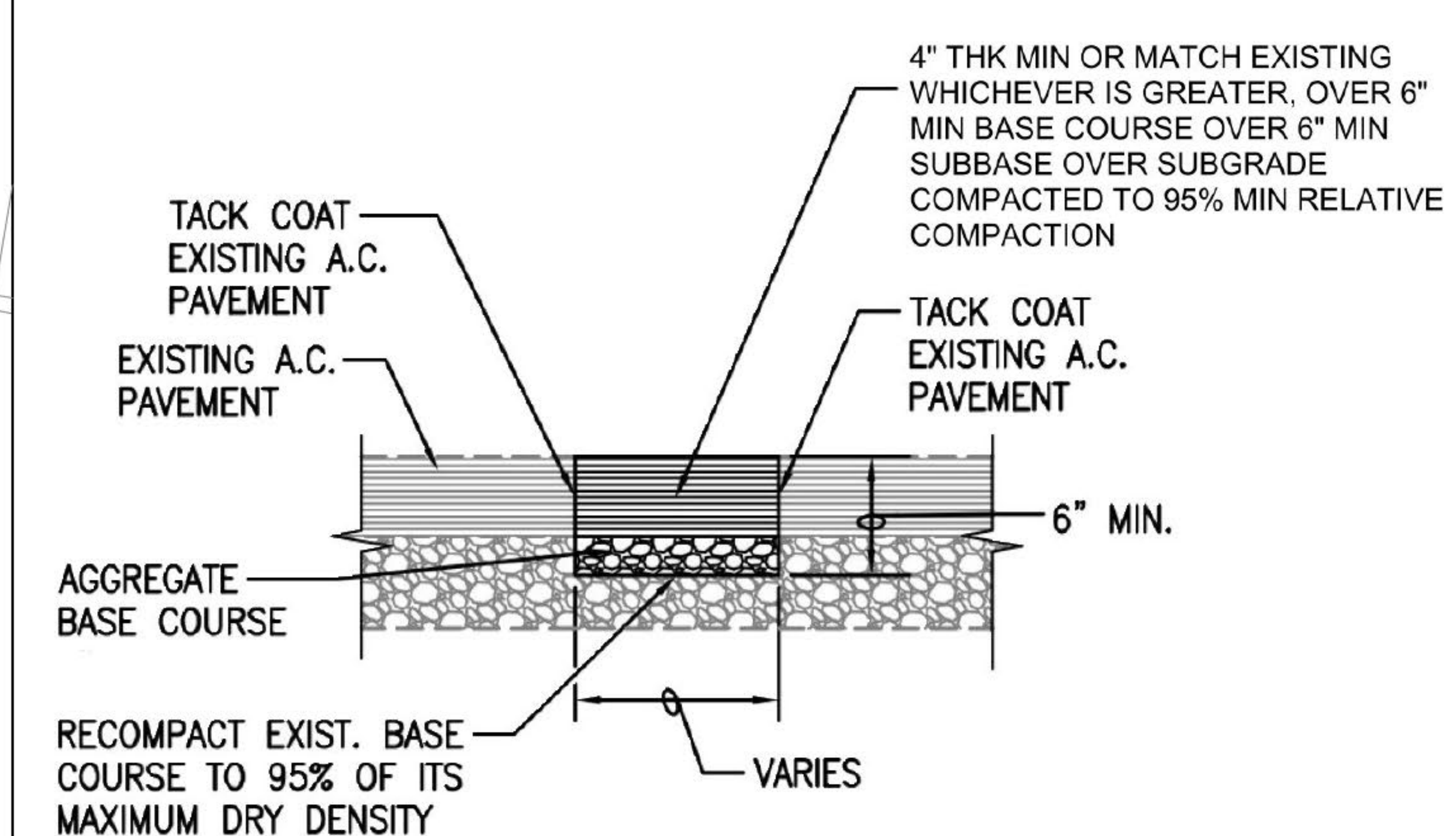


EXCAVATION SITE PLAN

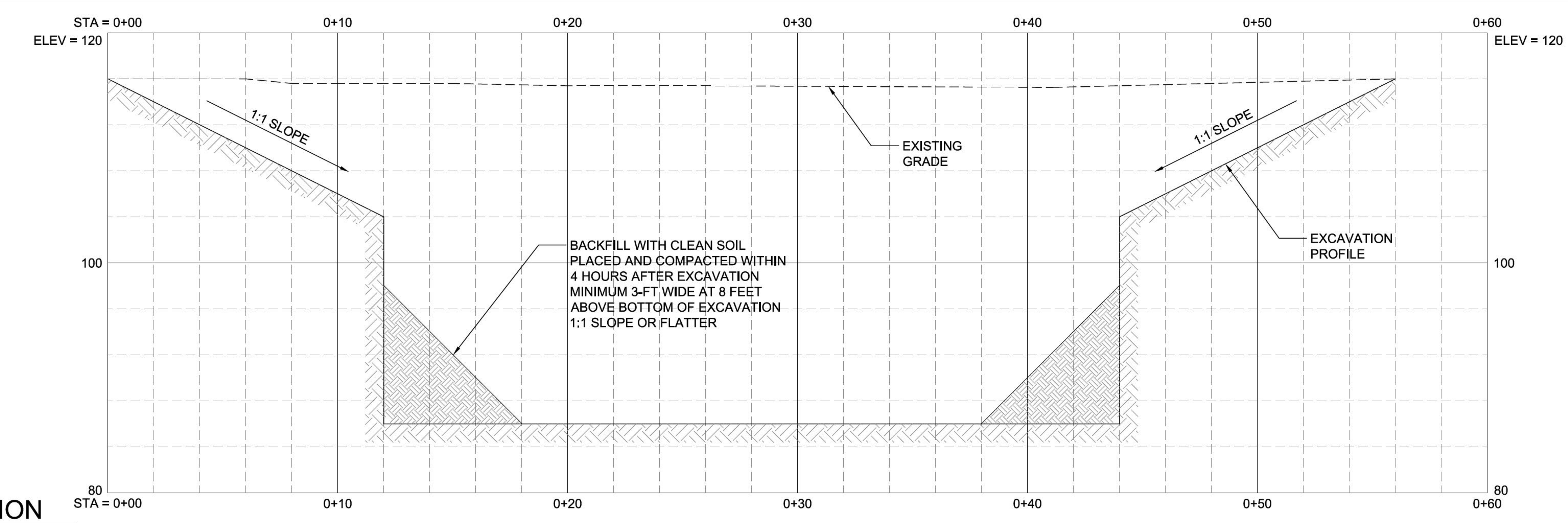
0' 4' 8' 16' 24'
1/8" = 1'-0"



LOCATION MAP



1 / 001 PAVEMENT REPAIR DETAIL
SCALE: NONE



2 / 001 SECTION
SCALE: AS SHOWN

GENERAL NOTES

A. ALL WORK SHALL CONFORM TO THE GOVERNING BUILDING CODES OF THE C & C OF HONOLULU.

B. CONTRACTOR SHALL CAREFULLY STUDY ALL OF THE CONTRACT DOCUMENTS AND SHALL VERIFY ALL EXISTING SITE CONDITIONS WITH CONSTRUCTION DOCUMENTS PRIOR TO PROCEEDING WITH ANY PORTION OF THE WORK AND TO NOTIFY THE OWNER OF ANY VARIANCES.

C. CONTRACTOR TO VERIFY ALL DIMENSIONS IN FIELD.

D. ANY CONDITIONS, MATERIALS, DEVICES OR DETAILS NOT SPECIFICALLY SHOWN ON THE DRAWINGS SHALL BE CLARIFIED WITH THE DESIGNER BEFORE CONSTRUCTION INSTALLATION OR APPLICATION. GENERAL SUBCONTRACTORS SHALL COMPLY WITH ALL GOVERNING CODES, BUILDING REGULATIONS OF FEDERAL, STATE, CITY AND COUNTY, WHICHEVER GOVERNS THE CONSTRUCTION WORK.

E. CONTRACTOR SHALL PROTECT ADJOINING LAND, BUILDING, AND OTHER IMPROVEMENTS SITUATED.

F. PROVIDE ALL CAULKING AND WATERPROOFING NECESSARY TO OBTAIN COMPLETE WEATHERPROOFING AND WEATHER TIGHT CONSTRUCTION.

G. ALL MATERIAL CALLED OUT ON DRAWINGS ARE TO BE NEW UNLESS OTHERWISE NOTED.

H. FIRE SAFETY DURING CONSTRUCTION, ALTERATION OR DEMOLITION SHALL BE IN ACCORDANCE WITH 2012 NFPA 1.

I. EXCAVATION AND BACKFILL SHALL BE OBSERVED BY A LICENSED GEOTECHNICAL ENGINEER.

J. NO STOCKPILE WITHIN 15 FEET AROUND THE EXCAVATION.

SGD Engineering
818 Sheridan St, Ste 209, Honolulu, HI 96814
Tel: (808) 428-0914 Email: info@sgdse.com

PROJECT TITLE: REDHILL EVCATION
PORTION OF AIEA AND HALAWA
AIEA, HAWAII 96701
TMK: (1) 9-9-010-006 (POR) & 050 (POR)

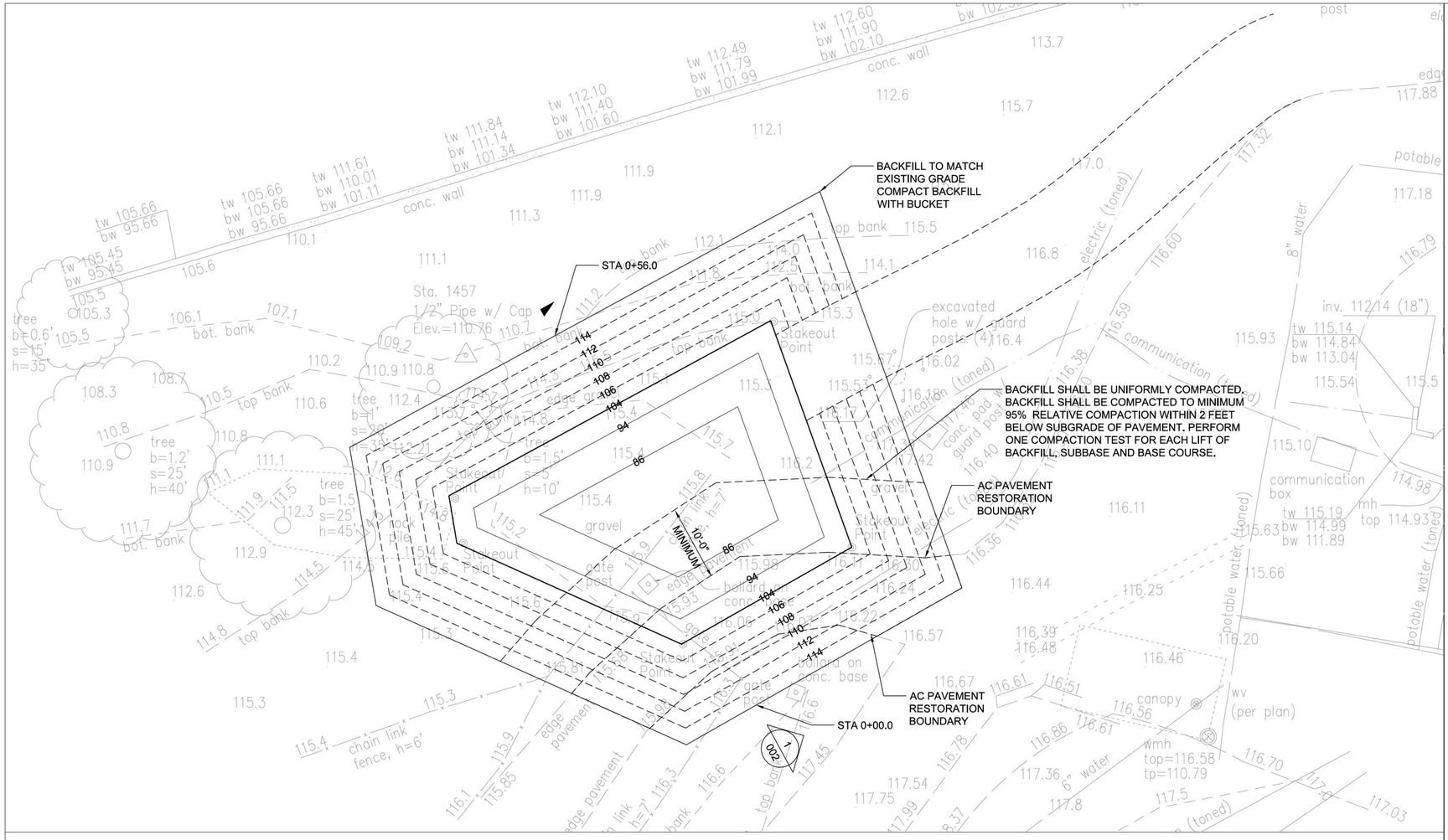
LONG CHEN
LICENSED PROFESSIONAL ENGINEER
No. 14321-S
HAWAII U.S.A.

SHEET TITLE: SITE PLAN AND DETAILS

REVISIONS:
DATE :
DATE :
DATE :
DATE :

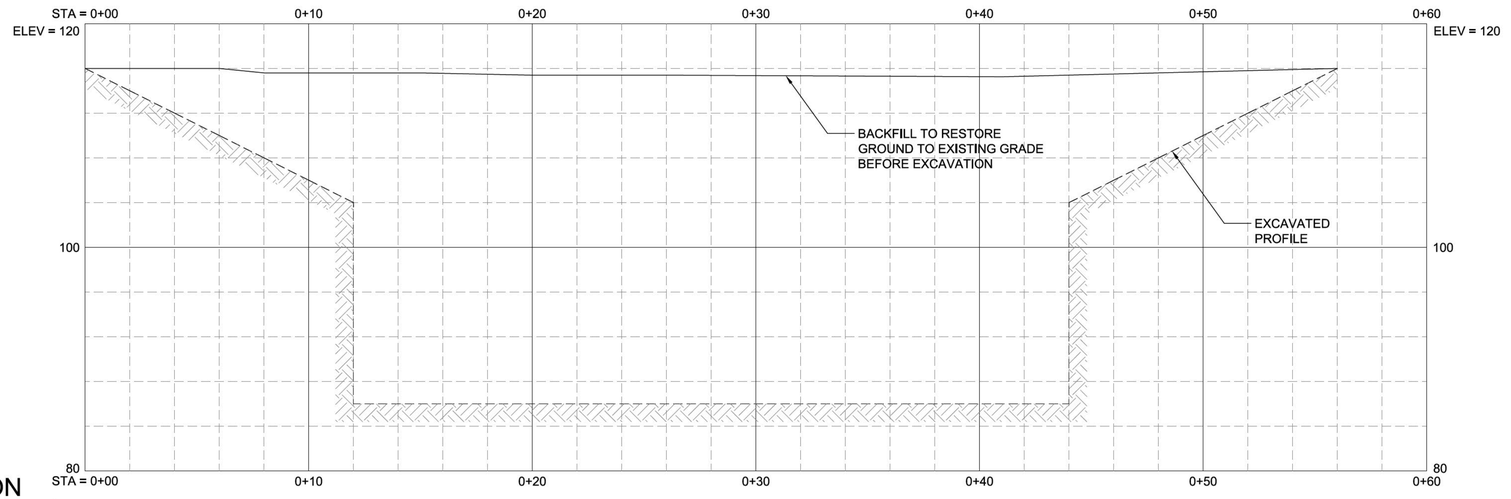
DESIGNED : SGD ENGR
DRAWN : SGD ENGR
CHECKED : SGD ENGR
SCALE : AS SHOWN

SHEET
001
SHEET 1 OF 2



BACKFILL SITE PLAN

O' 4' 8' 16' 24'
1/8" = 1'-0"



1/002 SECTION
SCALE: AS SHOWN

SGD Engineering
818 Sheridan St, Ste 209, Honolulu, HI 96814
Tel: (808) 428-9914 Email: lnfo@sgdse.com

PROJECT TITLE :
REDHILL EVCATION
PORTION OF AIEA AND HALAWA
AIEA, HAWAII 96701
TMK: (1) 9-9-010-006 (POR) & 050 (POR)

LONG CHEN
LICENSED PROFESSIONAL ENGINEER
No. 14321-S
HAWAII, U.S.A.

THIS WORK WAS PREPARED BY ME OR UNDER MY SUPERVISION I ATTEST TO THE CONSTRUCTION WILL BE DONE UNDER MY OBSERVATION.
4-30-2024
SIGNATURE DATE
EXPIRATION DATE
"OBSERVATION OF CONSTRUCTION" SHALL BE AS DEFINED IN CHAPTER 115, HAWAII ADMINISTRATIVE RULES FOR PROFESSIONAL ENGINEERS, ARCHITECTS, SURVEYORS, AND LANDSCAPE ARCHITECTS, STATE OF HAWAII, SUBCHAPTER 1, SECTION 16-115-C, DEFINITIONS, EFFECTIVE 9/29/76.

SHEET TITLE :
BACKFILL PLAN AND PROFILE

REVISIONS:
DATE :
DATE :
DATE :
DATE :

DESIGNED : SGD ENGR
DRAWN : SGD ENGR
CHECKED : SGD ENGR
SCALE : AS SHOWN

SHEET
002
SHEET 2 OF 2

LC
002_220202_FILL.DWG
08/17/2022 10:09

Appendix B: Backfill Certification and Specifications

Grace Pacific LLC
A SUBSIDIARY OF ALEXANDER & BALDWIN, INC.
G P Roadway Solutions • GLP Asphalt • Maui Paving

LABORATORY ANALYSIS OF BASALT 1.5 " SELECT BORROW (0241)

Company: **Pineridge Farms Inc.**

Project: **Red Hill Project**

Attention: [redacted]

Quarry: Makakilo

Sampled From: Storage Stockpile

Sampled By: [redacted]

Sieved By: [redacted]

AVERAGE GRADATION

Sq. Mesh Sieve #	Sieve Size (mm)	HDOT 703.17-1 Spec.	ASTM C136 % Passing
6"	152.4		
5"	127.0		
4"	101.6		
3"	76.1		
2 1/2"	63.5	100	100
2"	50.8		100
1 1/2"	38.1		99
1"	25.4		86
3/4"	19.0		76
1/2"	12.7		61
3/8"	9.51		55
No. 4	4.76	20-60	40
No. 8	2.38		30
No.10	2.00		29
No.16	1.19		24
No.20	0.841		
No. 30	0.595		20
No. 40	0.420		19
No. 50	0.297		17
No. 80	0.177		
No. 100	0.149		14
No. 200	0.074	0-15	11.1
ASTM D422	0.006		

PHYSICAL TEST RESULTS

SPEC		TEST STANDARD	ACTUAL
	Absorption:	ASTM C121	
	CBR:	ASTM D1883	84.9 @ 0.1"
	Clay Lumps & Friable Particles	ASTM C142	0.0
	Durability Index	ASTM D3744	98.0
	Flat & Elongates:	ASTM D4791	0.0
	LA Abrasions 500 Revolutions:	ASTM C131	28.5
	Liquid Limits:	ASTM D4318	CNBD-0
	Proctor Maximum Dry Density:	ASTM D1557	128.1 pcf
	Moisture Content %:	ASTM C566	
	Optimum Moisture:	ASTM D1557	12.1
	Organic Impurities:	ASTM C40	Lighter than standard
	Organic Matter:	ASTM D2974	
	Permeability:	ASTM D5084	
	Ph Analysis:	ASTM D4972	
<10	Plasticity Index:	ASTM D4318	NON-PLASTIC
	Resistivity (minimum):	AASHTO T288	
>25	Sand Equivalent:	ASTM D2419	41
	Soil Classification:	ASTM D2487	GP-GM
	Soundness:	ASTM C88	3.6
	Specific Gravity Fine :	AASHTO T84	
	Specific Gravity Coarse:	AASHTO T85	
	Expansion:	ASTM D1883	0.0
	Specification Compliance:	SSPWC 1986	Sec 30
	Unit Weight Ton/cubic yd	ASTM C29	1.40

NOTE: **DISCLAIMER: Proctor & optimum moisture is for general use only.**

It does NOT reflect actual material purchased. YOU must test the actual material

Revisions 08/10/2022 - Soil Classification GP-GM

[redacted]
QC Supervisor

August 18, 2022
Date

Grace Pacific LLC
A SUBSIDIARY OF ALEXANDER & BALDWIN, INC.
G P Roadway Solutions • GLP Asphalt • Maui Paving

LABORATORY ANALYSIS OF BASALT 3A FINE (0102-301)

Company: **Pineridge Farms**

Quarry: Makakilo

Project: **Red Hill**

Sampled From: Storage Stockpile

Sampled By: [redacted]

Attn: [redacted]

Sieved By: [redacted]

AVERAGE GRADATION

Sq. Mesh Sieve #	Sieve Size (mm)		ASTM C136 % Passing
6"	152.4		
5"	127.0		
4"	101.6		
3"	76.1		
2 1/2"	63.5		
2"	50.8		
1 1/2"	38.1		
1"	25.4		100
3/4"	19.0		100
1/2"	12.7		54
3/8"	9.51		21
No. 4	4.76		3
No. 8	2.38		2
No.10	2.00		
No.16	1.19		2
No.20	0.841		
No. 30	0.595		2
No. 40	0.420		
No. 50	0.297		1
No. 80	0.177		
No. 100	0.149		1
No. 200	0.074		1.2
ASTM D422	0.006		

PHYSICAL TEST RESULTS

SPEC		TEST STANDARD	ACTUAL
	Absorption:	ASTM C121	1.3
	CBR:	AASHTO T193	
	Clay Lumps & Friable Particles	ASTM C142	0.0
	Flat & Elongates:	ASTM D4791	0.0
<40	LA Abrasions 500 Revolutions:	ASTM C131	14.6
	Liquid Limits:	AASHTO T89	
	Maximum Dry Density:	AASHTO T180	
	Moisture Content %:	ASTM C566	
	Optimum Moisture:	AASHTO T180	
	Organic Matter:	ASTM D2974	
	Permeability:	ASTM D5084	
	Ph Analysis:	ASTM D4972	
	Plasticity Index:	AASHTO T90	
	Resistivity (minimum):	AASHTO T288	
	Sand Equivalent:	AASHTO T176	
	Soil Classification:	ASTM D2487	
<18	Soundness:	ASTM C88	1.9
	Specific Gravity Fine :	AASHTO T84	
	Specific Gravity Coarse:	AASHTO T85	2.932
	Swell Factor:	AASHTO T193	
	Specification Compliance:	ASTM C33	Size No.67
	Rodded Unit Weight	ASTM C29	

NOTE:

[redacted]
QC Supervisor

August 19, 2022
Date

Grace Pacific LLC
A SUBSIDIARY OF ALEXANDER & BALDWIN, INC.
G P Roadway Solutions • GLP Asphalt • Maui Paving

LABORATORY ANALYSIS OF BASALT 3B FINE (0261)

Company: **Pineridge Farms Inc.**

Quarry: Makakilo

Project: **Red Hill Project**

Sampled From: Storage Stockpile

Sampled By: [redacted]

Attention: [redacted]

Sieved By: [redacted]

AVERAGE GRADATION

Sq. Mesh Sieve #	Sieve Size (mm)	HDOT 703.16 Spec.	AASHTO T27 % Passing
6"	152.4		
5"	127.0		
4"	101.6		
3"	76.1		
2 1/2"	63.5		
2"	50.8		
1 1/2"	38.1		
1"	25.4	100	100
3/4"	19.0	90-100	90
1/2"	12.7		50
3/8"	9.51	20-55	29
No. 4	4.76	0-10	6
No. 8	2.38	0-5	5
No.10	2.00		
No.16	1.19		
No.20	0.841		
No. 30	0.595		
No. 40	0.420		
No. 50	0.297		
No. 80	0.177		
No. 100	0.149		3
No. 200	0.074		2.7
ASTM D422	0.006		

PHYSICAL TEST RESULTS

SPEC		TEST STANDARD	ACTUAL
	Absorption:	ASTM C121	3.8
	CBR:	AASHTO T193	
	Clay Lumps & Friable Particles	ASTM C142	0.0
	Flat & Elongates:	ASTM D4791	0.0
<40	LA Abrasions 500 Revolutions:	AASHTO T96	20.0
	Liquid Limits:	AASHTO T89	
	Maximum Dry Density:	AASHTO T180	
	Moisture Content %:	ASTM C566	
	Optimum Moisture:	AASHTO T180	
	Organic Matter:	ASTM D2974	
	Permeability:	ASTM D5084	
	Ph Analysis:	ASTM D4972	
	Plasticity Index:	AASHTO T90	
	Resistivity (minimum):	AASHTO T288	
	Sand Equivalent:	AASHTO T176	
	Soil Classification:	ASTM D2487	
	Soundness:	ASTM C88	1.6
	Specific Gravity Fine :	AASHTO T84	
	Specific Gravity Coarse:	AASHTO T85	2.626
	Swell Factor:	AASHTO T193	0.0
	Specification Compliance:	HAA	703.16
	Unit Weight Tons/CY	ASTM C29	1.39
	Durability Index:	ASTM D3744	98.00

NOTE: **Basalt 3B fine aggregate submittal is in compliance with HSS 703.16**

[redacted]
QC Supervisor

August 19, 2022
Date

Grace Pacific LLC
A SUBSIDIARY OF ALEXANDER & BALDWIN, INC.
G P Roadway Solutions • GLP Asphalt • Maui Paving

September 21, 2021

Subject: Clean Material Certification for Makakilo Quarry

To whom it may concern,

This notice serves to certify that the aggregate materials from the Grace Pacific Makakilo Quarry do not contain hazardous quantities of contaminants. This statement applies to virgin aggregate products, as well as recycled aggregate products.

Grace Pacific's mining and processing are done in a manner that ensures the aggregates do not come into contact with any type of fuels, oils, pesticides, herbicides, or other hazardous substances.

In accordance with 40 CFR Part 112 Grace Pacific prepares, updates, and complies with the Makakilo Quarry Spill Prevention Control and Countermeasure Plan (SPCC), March 13, 2015. The SPCC describes oil handling operations, spill prevention practices, discharge or drainage, controls, response measures to spills of oils or other potential pollutants, and the Facility's personnel, equipment and resources. This requirement is promulgated in the Clean Water Act (CWA).

A Phase 1 Environmental Site Assessment dated October 9, 2009 of the Makakilo Quarry site has been conducted. No recognized environmental conditions or potentially significant environmental conditions were observed in the active mining areas or the aggregate processing equipment/areas.

The aggregate recycling operation at the Makakilo Quarry is managed under a solid waste permit issued by the Hawaii Department of Health. Incoming materials are screened for potential contaminants to ensure the safety of the recycled aggregate products.

If you have any questions regarding this information, please contact me at (808) 674-8383.

Sincerely,

[redacted]

[redacted]
Environmental Compliance Manager

Appendix C: Coconut Rhinoceros Beetle Identification Sheet

Coconut Rhinoceros Beetle

Pests and Diseases of American Samoa

Number 8

American Samoa Community College
Community & Natural Resources
Cooperative Research & Extension 2005

Introduction. The coconut rhinoceros beetle, *Oryctes rhinoceros* (L.), has been a pest of coconuts and other palms in the South Pacific since its accidental introduction into Samoa from Sri Lanka in 1909. Rhinoceros beetle is mainly a pest of coconut and oil palms; but it also attacks other palm species.

Damage to coconut palm

Damage. Coconut rhinoceros beetle adults damage palms by boring into the center of the crown, where they injure the young, growing tissues and feed on the exuded sap. As they bore into the crown, they cut through the developing leaves. When the leaves grow out and unfold, the damage appears as V-shaped cuts in the fronds or holes through the midrib.

Adult

Life Cycle. Eggs are laid and larvae develop in decaying logs or stumps, piles of decomposing vegetation or sawdust, or other organic matter. Eggs hatch in 8-12 days, and larvae feed and grow for another 82-207 days before entering an 8-13 day nonfeeding prepupal stage. Pupae are formed in a cell made in the wood or in the soil beneath where the larvae feed. The pupal stage lasts 17-28 days. Adults remain in the pupal cell 17-22 days before emerging and flying to palm crowns to feed. The beetles are active at night and hide in feeding or breeding sites during the day. Most mating takes place at the breeding sites. Adults may live 4-9 months and each female lays 50-100 eggs during her lifetime.

Larva

Natural Enemies. Rhinoceros beetle eggs, larvae, pupae, and adults may be attacked by various predators, including pigs, rats, ants, and some beetles. They may also be killed by two important diseases: the fungus *Metarhizium anisopliae* and the *Oryctes* virus disease.

Larva infected with *Metarhizium anisopliae*

Published by the Division of Community and Natural Resources, American Samoa Community College, and issued in furtherance of Cooperative Extension Work Acts of May 8 and June 30, 1914, in cooperation with the U.S. Department of Agriculture Cooperative Extension Service. The American Samoa Community College Division of Community and Natural Resources prohibits discrimination in all its programs and activities on the basis of race, color, national origin, gender, religion, age, disability, political beliefs, sexual orientation, and marital or family status. For more information contact ASCC CNR at 684-699-1575.

Management. Rhinoceros beetles can be controlled by eliminating the places where they breed and by manually destroying adults and immatures.

- Chop and burn decaying logs or break them up and destroy any rhinoceros beetles developing inside.
- Cut stumps as close to the soil surface as possible.
- Dead, standing coconuts should be felled, chopped, dried, and burned.
- Rhinoceros beetles do not usually lay eggs in potential breeding sites that are obscured by growing vegetation. Vines or ground covers can be planted or allowed to grow over logs or stumps that cannot be destroyed.
- Piles of dead leaves or grass can be composted, used for mulch, burned, or spread on the ground in a thin layer.
- Compost piles should be maintained properly. When turning compost piles or applying compost to plants, destroy any rhinoceros beetles found. It takes longer for rhinoceros beetle larvae to develop than it takes to make compost, so properly maintained compost should not serve as a source of rhinoceros beetles.
- A hooked wire can be used to extract and destroy rhinoceros beetle adults feeding in palm crowns.

In many countries, the fungus *Metarhizium anisopliae* or the *Oryctes* virus are used to control the rhinoceros beetle. More recently a chemical attractant, ethyl-4-methyloctanoate, has been used in traps to attract and kill the beetles. Both *Metarhizium anisopliae* and the *Oryctes* virus are present and helping to reduce rhinoceros beetle populations in American Samoa; however, these pathogens and the attractant have not yet received approval from the United States Environmental Protection Agency for use as pesticides to control the rhinoceros beetle.

Females (left) tend to have shorter “horn” and fuzzy posterior.

Breeding site in decaying leaves.

Breeding site in coconut log.

References

Doane, R. W. 1913. The rhinoceros beetle (*Oryctes rhinoceros* L.) in Samoa. Journal of economic entomology 6: 437-442.

Gressit, J. L. 1953. The coconut rhinoceros beetle (*Oryctes rhinoceros*), with particular reference to the Palau Islands. Bernice P. Bishop Museum Bulletin No. 212. 157 p.

Swan, D. I. 1974. A review of the work on predators, parasites and pathogens for the control of *Oryctes rhinoceros* (Coleoptera: Scarabaeidae) in the Pacific area. Commonwealth Institute of Biological Control misc. pub. no. 7. 64 p.

Waterhouse, D. F. and Norris, K. R. 1987. Biological control: Pacific prospects. ACIAR, Melbourne.

Zelazny, B. 1979. Loss in coconut yield due to *Oryctes rhinoceros* damage. FAO Plant Protection Bulletin 27: 65-70.

This work was funded by a grant from USDA CSREES Integrated Pest Management Program. Prepared by Mark Schmaedick. *Metarhizium* photograph by Fred Brooks. For more information contact ASCC CNR at 684-699-1575.

Appendix D: Laboratory NELAP Certification

Accredited Laboratory

A2LA has accredited

PACE ANALYTICAL SERVICES

Mt. Juliet, TN

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) and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 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 April 2017).

Presented this 28th day of January 2022.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 1461.01
Valid to November 30, 2023

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

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

PACE ANALYTICAL NATIONAL
12065 Lebanon Road
Mt. Juliet, TN 37122
Rebecca King Phone: 615 773 9755

ENVIRONMENTAL

Valid To: November 30, 2023 Certificate Number: 1461.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories (QSM), and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, 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

Demands, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, HPLC, Hazardous Waste Characteristics Tests, Ion Chromatography, FIMS AA Cold Vapor, ICP, ICP-MS, MBAS, Misc. electrodes (pH, F, O), Titrimetry, Turbidity, Microbiology, Air, Bioassay, Gamma Spectrometry, Alpha Spectrometry, KPA, Liquid Scintillation, GFAA

*Solid Hazardous Waste methods, Microbiology, Air Testing and Bioassay are accredited to the requirements of the DoD ELAP and DOECAP.

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Metals				
Aluminum	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Antimony	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Arsenic	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Barium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Beryllium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B

5202 Presidents Court, Suite 220 | Frederick, MD 21703-8515 | Phone: 301 644 3248 | Fax: 240 454 9449 | www.A2LA.org

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Boron	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Cadmium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Calcium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Chromium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Cobalt	EPA 200.7	EPA 200.7	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Copper	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Iron	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Isotopic Uranium			EPA 6020, 6020A, 6020B	EPA 6020, 6020A, 6020B
Lead	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Lithium	EPA 200.7	EPA 200.7	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Magnesium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Manganese	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Mercury	EPA 245.1	EPA 245.1	EPA 7470 A	EPA 7471 A, B
Molybdenum	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Nickel	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Phosphorus	EPA 200.7	EPA 200.7	EPA 6010 B, C, D	EPA 6010 B, C, D
Potassium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Selenium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Silver	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Sodium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Strontium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Thallium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Tin	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Titanium	EPA 200.7	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Thorium	----------	EPA 200.8	EPA 6020, 6020A, 6020B	EPA 6020, 6020A, 6020B
Uranium	EPA 200.8	EPA 200.8	EPA 6020, 6020A, 6020B	EPA 6020, 6020A, 6020B
Vanadium	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Zinc	EPA 200.7 EPA 200.8	EPA 200.7 EPA 200.8	EPA 6010 B, C, D EPA 6020, 6020A, 6020B	EPA 6010 B, C, D EPA 6020, 6020A, 6020B
Metals Preparation	EPA 200.2	EPA 200.2	EPA 3015 A	EPA 3051 A EPA 3050 B
Nutrients				
Ammonia (as N)	----------	EPA 350.1 SM 4500 NH_3 B-2011 (prep) SM 4500 NH_3 G-2011	EPA 350.1 SM 4500 NH_3 B-2011 (prep) SM 4500 NH_3 G-2011	EPA 350.1 SM 4500 NH_3 B-2011 (prep) SM 4500 NH_3 G-2011
Kjeldahl Nitrogen	----------	EPA 351.2 SM 4500 N_{org} D-2011	----------	----------
Organic Nitrogen (as N)	----------	SM 4500 N_{org} D 2011	----------	----------

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Nitrate (as N)	EPA 300.0 SM 4110 B-2011 EPA 353.2 SM 4500-NO3 F-2011	EPA 300.0 SM 4110 B-2011 EPA 353.2 SM 4500 NO_3 F-2011 SM 4500 NO_3 F-2016	EPA 9056, 9056 A	EPA 9056, 9056 A
Nitrate-nitrite (as N)	EPA 300 .0 SM 4110 B-2011 EPA 353.2 SM 4500 NO3 F-2011	EPA 300.0 SM 4110 B- 2011 EPA 353.2 SM 4500 NO_3 F-2011 SM 4500 NO_3 F-2016	EPA 9056, 9056 A	EPA 9056, 9056 A
Nitrite (as N)	EPA 300.0 EPA 353.2 SM 4110 B-2011 SM 4500 NO^3F-2011	EPA 300.0 EPA 353.2 SM 4110 B-2011 SM 4500 NO3 F-2011	EPA 9056, 9056 A	EPA 9056, 9056 A
Orthophosphate (as P)	EPA 365.2 SM 4500 P E-2011	EPA 365.2 SM 4500 P E-2011	----------	EPA 9056, 9056 A
Total Phosphorus	----------	EPA 365.1 EPA 365.4 SM 4500 P H-2011 (prep) SM 4500 P B-2011 (prep)	----------	----------
Anion Preparation	----------	----------	----------	Prep: 300 Rv 2.1
Demands				
Biological Oxygen Demand	----------	SM 5210 B-2011 SM 5210 B-2016	----------	----------
Carbonaceous BOD	----------	SM 5210 B-2011 SM 5210 B-2016	----------	----------
Chemical Oxygen Demand	----------	EPA 410.4 SM 5220 D-2011	----------	----------
Dissolved Organic Carbon	SM 5310 C-2011	SM 5310 B-2011 SM 5310 B-2014	EPA 9060 A	
Total Organic Carbon	SM 5310 C-2011	SM 5310 B-2011 SM 5310 B-2014	EPA 9060 A	Walkley Black Mod.
Dissolved Oxygen	----------	SM 4500 O G-2016	----------	----------
Dissolved Carbon	----------	SM 5310 B-2014	----------	----------
Inorganic Carbon	----------	SM 5310 B-2014	----------	----------
Extractable Organic Halides	----------	----------	----------	EPA 9023
Total Organic Halides	----------	SM 5320 B-2010	EPA 9020 B	EPA 9076
Wet Chemistry				

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
% Ash	----------	----------	----------	ASTM D5468 ASTM D482
Acidity	----------	SM 2310 B-2011	----------	----------
Alkalinity	SM 2320 B-2011	EPA 310.1 EPA 310.2 SM 2320 B-2011	----------	----------
Bromide	EPA 300.0 SM 4110 B-2011	EPA 300.0 SM 4110 B-2011	EPA 9056, 9056 A	EPA 9056, 9056 A
BTU	----------	----------	----------	ASTM D240
Carbon Dioxide	SM 4500– CO2 D – 2011	SM 4500-CO2 D-2011		
Chlorite	EPA 300.1	EPA 300.1	EPA 300.1	EPA 300.1
Chlorate	EPA 300.0 EPA 300.1	EPA 300.0 EPA 300.1	EPA 9056, 9056A EPA 300.1	EPA 9056, 9056A EPA 300.1
Chloride	EPA 300.0 SM 4110 B – 2011	EPA 300.0 SM 4110 B-2011	EPA 9056, 9056 A	EPA 9056, 9056 A
Chlorine	SM 4500 Cl G – 2011	SM 4500 Cl G-2011	----------	----------
Color	SM 2120 B-2011	SM 2120 B-2011	----------	----------
Cyanide	EPA 335.4 SM 4500 CN B, C-2011 prep & (distillation) SM 4500 CN E-2011 (analysis)	EPA 335.4 SM 4500 CN B, C-2011 prep & (distillation) SM 4500 CN E-2011 (analysis) SM 4500 CN G-2016	EPA 9010 C EPA 9012 B	EPA 9012 B EPA 9013 EPA 9010 C
Cyanide Amenable to Chlorination	EPA 335.4 SM 4500 CN B-2011 (prep) SM 4500 CN G-2011	EPA 335.4 SM 4500 CN B - 2011 (prep) SM 4500 CN G-2011 SM 4500 CN G-2016	EPA 9010 C EPA 9012 B	EPA 9010 C EPA 9012 B
Density (Specific Gravity)	----------	SM 2710 F-2011	----------	----------
Total Dissolved Solids	SM 2540 C-2011	SM 2540 C-2011	----------	----------
Fractional Organic Carbon and Organic Matter	----------	----------	----------	ASTM D2974-07A
Fluoride	EPA 300.0 SM 4110 B-2011	EPA 300.0 SM 4110 B-2011 SM 4500 F B-2011 (prep) SM 4500 F C-2011	EPA 9056, 9056 A	EPA 9056, 9056 A

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Hardness	EPA 130.1 SM 2340 B-2011	EPA 130.1 EPA 200.7 SM 2340 B-2011	EPA 6010 B, C, D	----------
Hexavalent Chromium	EPA 218.6 SM 3500 Cr B-2011 SM 3500 Cr C-2011	EPA 218.6 M SM 3500 Cr B-2011 SM 3500 Cr C-2011	EPA 7196 A EPA 7199	EPA 7196 A Prep EPA 3060 A EPA 7199
Iron (Ferrous)	----------	SM 3500 Fe B-2011	----------	----------
MBAS (Surfactants)	SM 5540 C-2011	SM 5540 C-2011	----------	----------
Total Suspended Solids	SM 2540 D-2011	SM 2540 D-2011 SM 2540 D-2015	----------	----------
Oil and Grease	----------	EPA 1664 A EPA 1664 B	EPA 9070 A EPA 1664 A EPA 1664 B	EPA 9071 B
Oxidation Reduction Potential	SM 2580 B	SM 2580 B	SM 2580 B	SM 2580 B Modified
Perchlorate	EPA 314 Mod.	EPA 314 Mod.	EPA 314 Mod.	EPA 314 Mod.
pH	EPA 150.1 SM 4500-H B-2011	EPA 150.1 SM 4500-H B-2011	EPA 9040 B EPA 9040 C	EPA 9045 C EPA 9045 D
Phenols	EPA 420.1 EPA 420.4 SM 5530 D-2005	EPA 420.1 EPA 420.4 SM 5530 D-2005	EPA 9066	----------
Salinity	----------	SM 2520 B-2011	----------	----------
Settleable Solids	----------	SM 2540 F-2011 SM 2540 F-2015	----------	----------
Specific Conductance	EPA 120.1 SM 2510 B-2011	EPA 120.1 SM 2510 B-2011	EPA 9050 A	EPA 9050 A
Sulfide	----------	SM 4500-S2 D-2011	----------	EPA 9030 B EPA 9034
Sulfate	EPA 300.0 SM 4110 B-2011	EPA 300.0 SM 4110 B-2011	EPA 9056, 9056 A	EPA 9056, 9056 A
Sulfite	----------	4500-SO_3 B-2011	----------	----------
Total Solids	----------	SM 2540 B-2011 SM 2540 B-2015	----------	SM 2540 G (20^{th})
Turbidity	EPA 180.1 SM 2130 B-2011	EPA 180.1 SM 2130 B-2011	----------	----------
UV 254	SM 5910 B-2011	SM 5910 B-2011	----------	----------
Volatile Residue	----------	EPA 160.4 SM 2540 E-2011 SM 2540 E-2015	----------	----------
Microbiology*				
Enterococci	----------	Enterolert	----------	----------

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Fecal Coliform	----------	EPA 1681 Colilert-18 SM 9223 B-2016	EPA 1681	EPA 1681
Total Coliform	SM 9223 B-2004	SM 9223 B-2004 SM 9223 B-2016	----------	----------
Escherichia coli	SM 9223 B-2004	SM 9223 B-2004 SM 9223 B-2016	----------	----------
Legionella	Legiolert	Legiolert		
Heterotrophic Bacteria	SM 9215 B-2000	SM 9215 B-2000	----------	----------
Purgeable Organics (Volatiles)				
1,1,1,2-Tetrachloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1,1-Trichloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1,2-Trichloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1-Dichloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1-Dichloroethene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,1-Dichloropropene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2,3-Trichlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2,3-Trichloropropane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2,3-Trimethylbenzene	----------	EPA 624.1	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2,4-Trichlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2,4-Trimethylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2-Dibromo-3-chloropropane	EPA 524.2 EPA 504.1	EPA 624.1 SM 6200 B-2011 EPA 8011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2-Dibromomethane (EDB)	EPA 524.2 EPA 504.1	EPA 624.1 SM 6200 B-2011 EPA 8011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2-Dichlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
1,2-Dichloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,2-Dichloropropane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,3,5-Trichlorobenzene		EPA 624.1	EPA 8260 B, C, D	EPA 8260 B, C, D
1,3,5-Trimethylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,3-Butadiene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,3-Dichlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,3-Dichloropropane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,4-Dichlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1,4-Dioxane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D (Includes SIM)	EPA 8260 B, C, D (Includes SIM)
1-Butanol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
1-Methylnaphthalene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2,2,4-Trimethylpentane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2,2-Dichloropropane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Butanone (MEK)	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Chloroethyl Vinyl Ether	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Chlorotoluene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Hexanone	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Methylnaphthalene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
2-Nitropropane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
3,3-dimethyl-1-butanol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
4-Chlorotoluene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
4-Ethyltoluene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
4-Methyl-2-pentanone (MIBK)	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Acetone	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Acetonitrile	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Acrolein	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Acrylonitrile	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Allyl Chloride	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Benzene	EPA 524.2	EPA 602 EPA 624.1 SM 6200 B-2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
Bromobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260B, C, D	EPA 8260 B, C, D
Bromochloromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Bromodichloromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Bromoethane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Bromoform	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Bromomethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Carbon Disulfide	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Carbon Tetrachloride	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Chlorobenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Chloroethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Chloroform	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Chloromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Chloroprene (2-chloro-1,3-Butadiene)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
cis-1,2-Dichloroethene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
cis-1,3-Dichloropropene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
cis-1,4-Dichloro-2-Butene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Cyclohexane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Cyclohexanone	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Dibromochloromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Dibromomethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Dichlorodifluoromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Dicyclopentadiene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Diethyl Ether	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Diisopropyl Ether (DIPE)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Ethanol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D EPA 8015C	EPA 8260 B, C, D EPA 8015C
Ethyl Acetate	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Ethyl Methacrylate	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Ethylbenzene	EPA 524.2	EPA 602 EPA 624.1 SM 6200 B-2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
Ethyl-tert-butyl-ether (ETBE)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Gasoline Range Organics	----------	----------	EPA 8015 B, C, D EPA 8260 B, C, D NWTPH GX OA-1 WI GRO MADEP VPH MT VPH KS LRH AK 101 OK DEQ	EPA 8015 B, C, D EPA 8260 B, C, D NWTPH GX OA-1 WI GRO MADEP VPH MT VPH KS LRH AK 101 OK DEQ
C5-C6 Aliphatic Hydrocarbons	----------	----------	WA DOE VPH	WA DOE VPH
>C6-C8 Aliphatic Hydrocarbons	----------	----------	WA DOE VPH	WA DOE VPH
>C8-C10 Aliphatic Hydrocarbons	----------	----------	WA DOE VPH	WA DOE VPH
C8-C10 Aromatic Hydrocarbons	----------	----------	WA DOE VPH	WA DOE VPH

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Hexachlorobutadiene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Hexachloroethane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Iodomethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Iso-butyl Alcohol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Isopropanol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Ethylbenzene	EPA 524.2	EPA 602 EPA 624.1 SM 6200 B-2011	EPA 8021 B EPA 8260 B, C	EPA 8021 B EPA 8260 B, C
Ethyl Methacrylate	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C	EPA 8260 B, C
Ethyl-tert-butyl-ether (ETBE)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C	EPA 8260 B, C
42-Hexanone	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C	EPA 8260 B, C
Hexachlorobutadiene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C	EPA 8260 B, C
Isopropylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methacrylonitrile	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methanol	----------	----------	EPA 8015C	EPA 8015C
Methyl Acetate	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methyl Methacrylate	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methylcyclohexane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methylene Chloride	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Methyl-tert-butyl-ether (MTBE)	EPA 524.2	EPA 624.1 EPA 6200B- 2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
m-Xylene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Naphthalene	EPA 524.2	EPA 624.1 SM 6200B-2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
n-Butylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
n-Heptane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
n-Hexane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
n-Octane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
n-Propylbenzene	EPA 524.2	EPA 624.1 SM 6200B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
o-Xylene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Pentachloroethane	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
p-Isopropyltoluene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Propene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Propionitrile	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
p-Xylene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
sec-Butylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Styrene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
t-Amyl Alcohol	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
t-Butyl Formate	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
tert-Amyl ethyl ether	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Tert-Amyl-methyl-ether (TAME)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
tert-Butyl Alcohol (TBA)	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
tert-Butylbenzene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Tetrachloroethene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Tetrahydrofuran	EPA 524.2	EPA 624.1 SM 6200B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Toluene	EPA 524.2	EPA 602 EPA 624.1 SM 6200 B-2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
trans-1,2-Dichloroethene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
trans-1,3-Dichloropropene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
trans-1,4-Dichloro-2-Butene	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Trichloroethene	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Trichlorofluoromethane	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Trihalomethanes	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Vinyl Acetate	----------	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Vinyl Chloride	EPA 524.2	EPA 624.1 SM 6200 B-2011	EPA 8260 B, C, D	EPA 8260 B, C, D
Xylenes, total	EPA 524.2	EPA 602 EPA 624.1 SM 6200 B-2011	EPA 8021 B EPA 8260 B, C, D OK DEQ	EPA 8021 B EPA 8260 B, C, D OK DEQ
Purgeables Preparation	----------	----------	EPA 5030 C	EPA 5035 A EPA 3585
Extractable Organics (Semivolatiles)				
1,2,3,4-Tetrachlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,2,3,5-Tetrachlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,2,4,5-Tetrachlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,2,4-Trichlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,2-Dichlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,2-Diphenylhydrazine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,3,5-Trinitrobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,3-Dichlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,3-Dinitrobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,4-Dichlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,4-Dinitrobenzene		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,4-Dioxane	----------	EPA 625.1 SIM	EPA 8270 C, D, E SIM (Isotopic dilution)	EPA 8270 C, D, E SIM (Isotopic dilution)
1,4-Naphthoquinone	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1,4-Phenylenediamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1-Chloronaphthalene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
1-Methylnaphthalene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
1-Naphthylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,2-Oxybis(1-chloropropane)	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E

Environmental Analyses				
Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste	
			Aqueous*	Solid*
2,3,4,6-Tetrachlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4,5-Trichlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4,6-Trichlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4-Dichlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4-Dimethylphenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4-Dinitrophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,4-Dinitrotoluene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,6-Dichlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,6-Dinitrotoluene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2,6-Toluenediisocyanate		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Acetylaminofluorene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Chloronaphthalene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Chlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Methyl-4,6-Dinitrophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Methylnaphthalene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
2-Methylphenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Naphthylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Nitroaniline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Nitrodiphenylamine		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Nitrophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Picoline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
3,3'-Dichlorobenzidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
3,3-Dimethylbenzidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
3-Methylcholanthrene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
3-Methylphenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
3-Nitroaniline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4,4'-Methylenebis(2-chloroaniline)		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Aminobiphenyl	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Bromophenylphenylether	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Chloro-3-methylphenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Chloroaniline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Chlorophenyl Phenyl Ether	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Methylphenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Nitro Quinoline-1-Oxide	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Nitroaniline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
4-Nitrophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
5-Nitro-2-Toluidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
7,12-Dimethylbenz (a)anthracene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
7H-Dibenzo(c,g)carbazole	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Acenaphthene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Acenaphthylene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Acetophenone	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Alpha Terpineol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Aniline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Anthracene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Aramite	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Atrazine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzaldehyde	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzal Chloride	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzenethiol		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzo (a) Anthracene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM	EPA 8270 C, D, E (Includes SIM)
Benzo (a) Pyrene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Benzo (b) Fluoranthene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Benzo (ghi) Perylene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Benzo (k) Fluoranthene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Benzo(j)fluoranthene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzoic Acid	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzotrichloride	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzyl Alcohol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Benzyl Chloride	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Biphenyl (1,1-)	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Bis (2-chloroethoxy) methane	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Bis (2-chloroethyl) Ether	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Bis (2-ethylhexyl) phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Butyl benzyl phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Caprolactam	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Carbazole	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Chlorobenzilate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Chrysene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
cis-Diallate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
cis-Isosafrole	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Dibenzo (a,e) pyrene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dibenzo (a,h) acridine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dibenzo (a,h) anthracene	----------	EPA 625.1	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Dibenzo (a,i) pyrene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dibenzo (a,j) acridine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dibenzofuran	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Diesel Range Organics / Oil Range Organics / Residual Range Organics	----------	----------	EPA 8015 B, C, D FL PRO OA2 TN EPH TX 1005 WI DRO NWTPH Dx NWTPH ID MADEP EPH MT EPH NJ EPH KS MRH-HRH 8270C DRO MO AK 102 AK 103 EPA 8270 C, D, E OK DEQ TX 1006	EPA 8015 B, C, D FL PRO OA2 TN EPH TX 1005 WI DRO NWTPH Dx NWTPH ID MADEP EPH MT EPH NJ EPH KS MRH-HRH 8270C DRO MO AK 102 AK 103 EPA 8270 C, D, E OK DEQ TX 1006
C10-C12 Aliphatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C12-C16 Aliphatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C16-C21 Aliphatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C21-C34 Aliphatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C10-C12 Aromatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C12-C16 Aromatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C16-C21 Aromatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
C21-C34 Aromatic Hydrocarbons	----------	----------	WA DOE EPH	WA DOE EPH
Diethyl Phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dimethoate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Dimethyl Phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Di-n-butyl Phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Di-n-octyl Phthalate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Dinoseb	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Diphenyl Ether	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Diphenyl Ketone (Benzophenone)	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Diphenylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Disulfoton	----------	----------	EPA 8270 C, D, E	EPA 8270 C, D, E
2-Ethosyethanol	----------	EPA 8015B, 8015C Rv3, 8015D	EPA 8015B, 8015C Rv3, 8015D	EPA 8015B, 8015C Rv3, 8015D
Ethylene Glycol	----------	----------	EPA 8015 B, C, D	EPA 8015 B, C, D
Ethyl Methanesulfonate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Famphur	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Fluoranthene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Fluorene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Hexachlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hexachlorobutadiene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hexachlorocyclopentadiene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hexachloroethane	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hexachlorophene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hexachloropropene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Hydroquinone	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Indene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Indeno (1,2,3-cd) Pyrene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Isodrin	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Isophorone	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Kepone	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Methapyrilene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Methyl Methanesulfonate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Mirex		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Naphthalene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
n-Decane	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Nitrobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosodiethylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosodimethylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitroso-di-n-butylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosodi-n-propylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosodiphenylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosomethylethylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosomorpholine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
n-Nitrosopiperidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Nitrosopyrrolidine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
n-Octadecane	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
O,O,O-Triethyl phosphorothioate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
o-Toluidine (2-Methylaniline)	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Parathion Ethyl	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Parathion Methyl	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
p-Dimethlaminoazobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pentachlorobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pentachloroethane	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pentachloronitrobenzene	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pentachlorophenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Phenacetin	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Phenanthrene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Phenol	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Phorate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Phthalic Anhydride		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Polynuclear Aromatic Hydrocarbons	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Propylene Glycol	----------	EPA 8015 B, C, D	EPA 8015 B, C, D	EPA 8015 B, C, D
Pronamide	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pyrene	----------	EPA 625.1 (Includes SIM)	EPA 8270 C, D, E (Includes SIM)	EPA 8270 C, D, E (Includes SIM)
Pyridine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Quinoline	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Safrole	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Sulfotepp	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Tetra Ethyl Lead			EPA 8270 C, D, E	EPA 8270 C, D, E
Thionazin	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
trans-Diallate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
trans-Isosafrole	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Triethyl Glycol		EPA 8015B, 8015C Rv 3, 8015D	EPA 8015B, 8015C Rv 3, 8015D	EPA 8015B, 8015C Rv 3, 8015D
Triclosan		EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
tris-(2,3-dibromopropyl) phosphate	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
α,α-Dimethylphenethylamine	----------	EPA 625.1	EPA 8270 C, D, E	EPA 8270 C, D, E
Pesticides/Herbicides/ PCBs				
Atrazine	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B

Environmental Analyses				
Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste	
			Aqueous*	Solid*
Azinphos Methyl	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Bolstar	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Butachlor	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Carbophenothion	----------	----------	EPA 8141 B	EPA 8141 B
Chlorpyrifos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Coumaphos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Cyanazine	----------	----------	EPA 8141 B	EPA 8141 B
Demeton-O & -S	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Diazinon	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Dichlorvos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Dimethoate	----------	EPA 1657 Mod.	EPA 8141 B	EPA 8141 B
Disulfoton	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
EPN	----------	EPA 1657 Mod.	EPA 8141 B	EPA 8141 B
Ethion	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Ethoprop	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Famphur	----------	----------	EPA 8141 B	EPA 8141 B
Fensulfothion	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Fenthion	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Malathion	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Merphos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Mevinphos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Naled	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Parathion Ethyl	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Parathion Methyl	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Phorate	----------	----------	EPA 8141 B	EPA 8141 B
Phosmet	----------	----------	EPA 8141 B	EPA 8141 B
Ronnel	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Simazine	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Stirophos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Sulfotepp	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
TEPP	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Tetrachlorvinphos	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Tokuthion (Prothiofos)	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
Trichloronate	----------	EPA 1657 A Mod.	EPA 8141 B	EPA 8141 B
4,4'-DDD	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
4,4'-DDE	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
4,4'-DDT	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Aldrin	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
alpha-BHC	----------	EPA 608.3	EPA 8081 B	EPA 8081 B
beta-BHC	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Chlordane	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Chlordane (Alpha)	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Chlordane (Gamma)	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
delta-BHC	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Dieldrin	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B

Environmental Analyses				
Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste	
			Aqueous*	Solid*
Endosulfan I (alpha)	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Endosulfan II (beta)	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Endosulfan sulfate	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Endrin	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Endrin aldehyde	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Endrin ketone	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
gamma-BHC	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Heptachlor	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Heptachlor Epoxide	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Hexachlorobenzene	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Methoxychlor	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
Toxaphene	----------	EPA 608.3	EPA 8081 A, B	EPA 8081 A, B
PCB-1016 (Arochlor)	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1221	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1232	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1242	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1248	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1254	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1260	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1262	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
PCB-1268	----------	EPA 608.3	EPA 8082, 8082 A	EPA 8082, 8082 A
2,4,5-T	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
2,4,5-TP (Silvex)	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
2,4-D	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
2,4-DB	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
Dalapon	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
Dicamba	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
Dichloroprop	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
Dinoseb	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
MCPA	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
MCPP	----------	----------	EPA 8151 A, EPA 8321B 2007	EPA 8151 A, EPA 8321B 2007
Pentachlorophenol	----------	----------	EPA 8151 A	EPA 8151 A
Dibromoacetic Acid	EPA 552.2	----------	----------	----------
Dichloroacetic Acid	EPA 552.2	----------	----------	----------
Monobromoacetic Acid	EPA 552.2	----------	----------	----------

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Monochloroacetic Acid	EPA 552.2	----------	----------	----------
Trichloroacetic Acid	EPA 552.2	----------	----------	----------
Organic Preparation				
Microextraction	----------	----------	EPA 3511	----------
Separatory funnel	----------	----------	EPA 3510 C EPA 3510 C RVE	----------
Sonication	----------	----------	----------	EPA 3550 B, C
Soxtherm	----------	----------	----------	EPA 3541
Microwave	----------	----------	----------	EPA 3546
Clean up	----------	----------	EPA 3580 A EPA 3630 C EPA 3660 B EPA 3665 A	EPA 3580 A EPA 3630 C EPA 3660 B EPA 3665 A
Explosives				
1,3,5-Trinitrobenzene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
1,3-Dinitrobenzene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
2,4,6-Trinitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
2,4-Dinitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
2,6-Dinitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
2-Amino-4,6-dinitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
2-Nitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
3-Nitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
4-Amino-2,6-dinitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
4-Nitrotoluene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	----------	----------	EPA 8330 A, B	EPA 8330 A, B
Nitrobenzene	----------	----------	EPA 8330 A, B	EPA 8330 A, B
Nitroglycerine	----------	----------	EPA 8330 A, B	EPA 8330 A, B
Nitroguanidine	----------	----------	EPA 8330 A, B	EPA 8330 A, B
PETN (Pentaerythritol Tetranitrate)	----------	----------	EPA 8330 A, B	EPA 8330 A, B
RDX (Hexahydro-1,3,5-trinitro-1,3,5-triazine)	----------	----------	EPA 8330 A, B	EPA 8330 A, B
Tetryl (N-methyl-N,2,4,6-trinitrophenylnitramine)	----------	----------	EPA 8330 A, B	EPA 8330 A, B
Guanidine Nitrate	----------	----------	EPA 9056 A Mod.	EPA 9056 A Mod.
Nitrocellulose	----------	----------	EPA 353.2 Mod.	EPA 353.2 Mod.
8330A/8330B Prep	----------	----------	EPA 3535 A	EPA 3550 C
Hazardous Waste Characteristics				
Conductivity	----------	----------	EPA 9050 A	----------
Corrosivity	----------	----------	EPA 9045 C, D	EPA 9045 C, D

Environmental Analyses				
Parameter/Analyte	**Potable Water**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous*	**Solid***
Ignitability	----------	----------	EPA 1010 EPA 1010 A	EPA 1010 EPA 1010 A
Paint Filter Liquids Test	----------	----------	----------	EPA 9095 A, B
Toxicity Characteristic Leaching Procedure	----------	----------	----------	EPA 1311
Synthetic Leachate Procedure	----------	----------	----------	EPA 1312
Bioassay*				
Ceriodaphnia Dubia Survival and Reproduction Test	----------	EPA 1002	----------	----------
Fathead Minnow Larval Survival and Growth Test	----------	EPA 1000	----------	----------
Ceriodapnia – Acute	----------	EPA 2002	----------	----------
Fathead Minnow – Acute	----------	EPA 2000	----------	----------

Air Testing		
Parameter/Analyte	**Air**	**Nonpotable Water**
1,1,1,-Trichloroethane	TO-15, TO-15 SIM	----------
1,1,2,2-Tetrachloroethane	TO-15, TO-15 SIM	----------
1,1,2-Trichloroethane	TO-15, TO-15 SIM	----------
1,1-Dichloroethane	TO-15. TO-15 SIM	----------
1,1-Dichloroethene	TO-15, TO-15 SIM	----------
1,1-Difluoroethane	TO-15	----------
1,2,3-Trimethylbenzene	TO-15	----------
1,2,4-Trichlorobenzene	TO-15	----------
1,2,4-Trimethylbenzene	TO-15	----------
1,2-Dibromoethane	TO-15, TO-15 SIM	----------
1,2-Dichlorobenzene	TO-15	----------
1,2-Dichloroethane	TO-15	----------
1,2-Dichloropropane	TO-15, TO-15 SIM	----------
1,3,5-Trimethylbenzene	TO-15	----------
1,3-Butadiene	TO-15	----------
1,3-Dichlorobenzene	TO-15	----------
1,4-Bromofluorobenzene	TO-15, TO-15 SIM	
1,4-Dichlorobenzene	TO-15, TO-15 SIM	----------
1,4-Dioxane	TO-15	----------
2,2,4-Trimethylpentane	TO-15	----------
2-Chlorotoluene	TO-15	----------
2-Methylnaphthalene	TO-15	
4-Ethyltoluene	TO-15	----------
Acetaldehyde	TO-15	
Acetone	TO-15	----------

Air Testing		
Parameter/Analyte	**Air**	**Nonpotable Water**
Acetonitrile	TO-15	
Acrolein	TO-15	
Acrylonitrile	TO-15	
Allyl Chloride	TO-15	----------
Benzene	TO-15, TO-15 SIM	----------
Benzyl Chloride	TO-15	----------
Bromodichloromethane	TO-15	----------
Bromoform	TO-15	----------
Bromomethane	TO-15	----------
Carbon Disulfide	TO-15	----------
Carbon Tetrachloride	TO-15, TO-15 SIM	----------
Chlorobenzene	TO-15	----------
Chlorodifluoromethane	TO-15	----------
Chloroethane	TO-15, TO-15 SIM	----------
Chloroform	TO-15, TO-15 SIM	----------
Chloromethane	TO-15, TO-15 SIM	----------
cis-1,2-Dichloroethene	TO-15, TO-15 SIM	----------
cis-1,3-Dichloropropene	TO-15, TO-15 SIM	----------
Cyclohexane	TO-15	----------
Dibromochloromethane	TO-15	----------
Ethanol	TO-15	----------
Ethyl Acetate	TO-15	----------
Ethylbenzene	TO-15, TO-15 SIM	----------
Freon-11 (Trichlorofluoromethane)	TO-15	----------
Freon-113 (1,1,2-Trichlorotrifluoroethane)	TO-15	----------
Freon-114 (1,2-Dichlorotetrafluoroethane)	TO-15	----------
Freon-12 (Dichlorodifluoromethane)	TO-15	----------
Gasoline Range Organics	TO-15	----------
Hexachloro-l,3-Butadiene	TO-15	----------
Iodomethane	TO-15	
Isopropylbenzene	TO-15	----------
lsopropyl Alcohol	TO-15	----------
Methanol	TO-15	
Methyl Butyl Ketone (2-Hexanone)	TO-15	----------
Methyl Cyclohexane	TO-15	----------
Methyl Ethyl Ketone (2-Butanone)	TO-15	----------
Methyl Isobutyl Ketone (4-Methyl-2-pentanone)	TO-15	----------
Methyl Methacrylate	TO-15	----------
Methyl-tert-butyl-Ether	TO-15	----------

Air Testing		
Parameter/Analyte	**Air**	**Nonpotable Water**
Methylene Chloride	TO-15	----------
m-xylene	TO-15	----------
Naphthalene	TO-15	----------
n-Butane	TO-15	
n-Butylbenzene	TO-15	----------
n-Heptane	TO-15	----------
n-Hexane	TO-15	----------
n-Nonane	TO-15	
n-Pentane	TO-15	
n-Propylbenzene	TO-15	----------
o-xylene	TO-15	----------
Propylene	TO-15	----------
p-xylene	TO-15	----------
sec-Butylbenzene	TO-15	----------
Styrene	TO-15	----------
tert-Amyl-ethyl-ether	TO-15	----------
tert-Butyl-alcohol	TO-15	----------
tert-Butylbenzene	TO-15	----------
Tetrachloroethylene	TO-15, TO-15 SIM	----------
Tetrahydrofuran	TO-15	----------
Toluene	TO-15	----------
trans-1,2-Dichloroethene	TO-15, TO-15 SIM	----------
trans-1,3-Dichloropropene	TO-15, TO-15 SIM	----------
Trichloroethylene	TO-15, TO-15 SIM	----------
Vinyl acetate	TO-15, TO-15 SIM	----------
Vinyl bromide	TO-15	----------
Vinyl Chloride	TO-15, TO-15 SIM	----------
Xylenes (Total)	TO-15	----------
Acetylene	EPA 8015 M	RSK-175
Butane	EPA 8015 M	RSK-175
Ethane	EPA 8015 M	RSK-175
Ethene	EPA 8015 M	RSK-175
Isobutane	EPA 8015 M	RSK-175
Methane	EPA 8015 M	RSK-175
Propane	EPA 8015 M	RSK-175
Carbon Dioxide	ASTM D1946	----------
Carbon Monoxide	ASTM D1946	----------
Methane	ASTM D1946	----------
Oxygen	ASTM D1946	----------

Radiochemistry		
Parameter/Analyte	**Nonpotable Water**	**Solid**

Gross Alpha	EPA 900 EPA 9310 ASTM D7283-13 (Liquid Scintillation)	EPA 900 Mod. EPA 9310 Mod. ASTM D7283-13 (Liquid Scintillation)
Gross Beta	EPA 900 EPA 9310 ASTM D7283-13 (Liquid Scintillation)	EPA 900 Mod. EPA 9310 Mod. ASTM D7283-13 (Liquid Scintillation)
Radium-228	EPA 904 EPA 9320	EPA 904 Mod. EPA 9320 Mod.
Total Strontium	EPA 905	EPA 905 Mod.
Strontium-89	EPA 905	EPA 905 Mod.
Strontium-90	EPA 905	EPA 905 Mod,
Tritium	EPA 906.0	EPA 906.0 Mod.
Carbon-14	EPA C-01	EPA C-01 Mod.
Radium-226	SM 7500-Ra B SM 7500 Ra B Mod. EPA 903 EPA 9315	SM 7500 Ra B Mod. EPA 903 EPA 9315
Total Alpha Radium	SM 7500-Ra B	SM 7500-Ra B Mod.
Radon-222	SM 7500-Rn B	
Am-241	EPA 907 Mod.	EPA 907 Mod.
Am-242/243	EPA 907 Mod.	EPA 907 Mod.
Cm-242	EPA 907 Mod.	EPA 907 Mod.
Cm-243/244	EPA 907 Mod.	EPA 907 Mod.
Radiochemistry		
Parameter/Analyte	**Nonpotable Water**	**Solid**
Cm-244	EPA 907 Mod.	EPA 907 Mod.
Cm-245/246	EPA 907 Mod.	EPA 907 Mod.
I-129	EPA 902 M	EPA 902 M
Ni-63	CHEM-TP-Ni.1 Modified	CHEM-TP-Ni.1 Modified
Np-237	EPA 907 Mod.	EPA 907 Mod.
Pb-210	ASTM D7535-09	ASTM D7535-09
Po-210	DOE HASL 300 Po-2 RC	DOE HASL 300 Po-2 RC
Pu-238	EPA 907 Mod.	EPA 907 Mod.
Pu-239	EPA 907 Mod.	EPA 907 Mod.
Pu-239/240	EPA 907 Mod.	EPA 907 Mod.
Th-227	LANL ER 200 Mod.	LANL ER 200 Mod.
Th-228	LANL ER 200 Mod.	LANL ER 200 Mod.
Th-230	LANL ER 200 Mod.	LANL ER 200 Mod.
Th-232	LANL ER 200 Mod.	LANL ER 200 Mod.
U-233/234	ASTM D3972	ASTM D3972 Mod.
U-235	ASTM D3972	ASTM D3972 Mod.
U-235/236	ASTM D3972	ASTM D3972 Mod.
U-238	ASTM D3972	ASTM D3972 Mod.
Total Uranium	ASTM D5174	ASTM D5174 Mod.
Gamma Spectrometry	EPA 901.1 HASL 300 Ga-01-R DOE 4.5.2.3	HASL 300 Ga-01-R DOE 4.5.2.3

Tc-99	TC-02-RC	TC-02-RC

Kentucky UST Program		
Parameter/Analyte	**Solid Hazardous Waste**	
	Aqueous	**Solid**
Toxicity Characteristic Leaching Procedure (Metals & Organics	----------	EPA 1311
TPH (Oil & Grease)	EPA 9070A EPA 1664A EPA 1664B	EPA 9071A
Metals		
Arsenic	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Barium	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Cadmium	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Chromium	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Lead	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Mercury	EPA 7470A	EPA 7471A EPA 7471B
Selenium	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Silver	EPA 6010B, 6010C EPA 6020, 6020A	EPA 6010B, 6010C EPA 6020, 6020A
Organics		
Gasoline Range Organics	EPA 8015 B, C, D	EPA 8015 B, C, D
Diesel Range Organics	EPA 8015 B, C, D	EPA 8015 B, C, D
Benzene	EPA 8260 B	EPA 8260 B
Ethyl Benzene	EPA 8260 B	EPA 8260 B
Toluene	EPA 8260 B	EPA 8260 B
Xylenes	EPA 8260 B	EPA 8260 B
TPH (Fingerprint)	EPA 8015 B, C, D	EPA 8015 B, C, D
Volatile Organic Compounds	EPA 8260 B	EPA 8260 B
Semi-Volatile Organics	EPA 8270 C	EPA 8270 C
PAH	EPA 8310 EPA 8270C	EPA 8310 EPA 8270C

Wyoming STR Program		
Parameter/Analyte	**Solid Hazardous Waste**	
	Aqueous	**Solid**
Metals		
Cadmium	EPA 6010 C	EPA 6010 C
Chromium	EPA 6010 C	EPA 6010 C
Lead	EPA 6010 C	EPA 6010 C
Purgeable Organics (Volatiles)		
Benzene	EPA 8260 B	EPA 8260 B
Diisopropyl Ether	EPA 8260 B	EPA 8260 B
Ethanol	EPA 8260 B	EPA 8260 B
Ethyl Benzene	EPA 8260 B	EPA 8260 B
Ethyl-t-butyl Ether	EPA 8260 B	EPA 8260 B
Gasoline Range Organics C6-C10	EPA 8015 C EPA 8260 B	EPA 8015 C EPA 8260 B
Methyl-t-butyl ether (MTBE)	EPA 8260 B	EPA 8260 B
Naphthalene	EPA 8260 B	EPA 8260 B
Toluene	EPA 8260 B	EPA 8260 B
t-Amyl-Methyl Ether	EPA 8260 B	EPA 8260 B
t-Butyl Alcohol	EPA 8260 B	EPA 8260 B
Xylenes, Total	EPA 8260 B	EPA 8260 B
1,2-Xylene	EPA 8260 B	EPA 8260 B
1,3-Xylene	EPA 8260 B	EPA 8260 B
1,4-Xylene	EPA 8260 B	EPA 8260 B
Extractable Organics		
Diesel Range Organics C10-C32	EPA 8015 C	EPA 8015 C

Appendix E: Laboratory SOPs

Document Information

Document Number: ENV-SOP-MTJL-0081 **Revision:** 05

Document Title: Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode

Department(s): SVOA

Date Information

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All Dates and Times are listed in: Central Time Zone

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All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0081

QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality	17 Jan 2022, 06:16:58 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Quality Analyst 3	13 Jan 2022, 03:03:23 PM	Approved

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STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, D, E & 625.1) Including Provisions for Analysis in SIM Mode
ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0080.

1.1 This method is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The lists of compounds that are routinely determined by this method are listed in Attachment II. This table represents a default list to be used in the absence of a project-specific list, which would take precedence. See section 13.4.

1.2 This method is used to quantitate most neutral, acidic and/or basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica column coated with a slightly polar methyl silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

1.3 In general, this method is not appropriate for the quantitation of multi-component analytes (i.e. Toxaphene, Chlordane, Aroclors, etc.) because of the limited sensitivity for those analytes; however when those analytes are identified using another analytical technique, this procedure is appropriate for confirmation pending sufficient analyte concentration is present in the extract.

1.4 Detection limits, sensitivity and optimum ranges of organic compounds vary with sample matrices, extraction technique, detector parameters, and model of GC/MS.

1.5 Qualifier ions are method specified and can be found in Attachment IV.

1.6 Use of this method is restricted to analysts who are knowledgeable in the interpretation of Mass Spectrometry and use of GC/MS systems.

1.7 The use of selected ion monitoring (SIM) is acceptable for applications requiring limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

1.8 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ENV-SOP-MTJL-0016. Updated MDL records are filed and stored on the intranet.

1.8.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM (see Attachment IX).

1.8.2 Lower Limit of Quantitation (LOQ) – For analyses performed per the requirements of Method 8000D, the LLOQ is established at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 2.10 and 10.4).

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STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, D, E & 625.1) Including Provisions for Analysis in SIM Mode
ISSUER: Pace National – Mt. Juliet, Tennessee

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Field samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample extraction technique. See appropriate SOPs for extraction and extract concentration methods. A measured volume or weight of sample is extracted using the appropriate extraction technique. Liquid samples are extracted at a reduced volume of around 100mls using EPA method 3510C (ENV-SOP-MTJL-0114). Large volume injection (LVI) extraction using EPA method 3511 that requires a smaller volume (usually 40mL) of field sample is also available for use where applicable. Soil analysis using the same technology can also be performed with extraction as noted in ENV-SOP-MTJL-0118 and no concentration performed on the extract. This process is termed throughout this SOP as non-concentrated soil. Solid samples can also be extracted traditionally using methylene chloride-acetone (1:1)) or with methylene chloride using the microwave process (ENV-SOP-MTJL-0118), where permitted. These extracts are denoted in this procedure using the terminology “concentrated soil” extracts.

2.2 The semi-volatile compounds are introduced into the GC/MS by directly injecting a volume of the sample extract into a gas chromatograph oven (GC) equipped with a narrow-bore fused-silica capillary column. The oven, containing the capillary column, is temperature and pressure programmed to separate the analytes by molecular composition. The capillary column transfers the eluting analytes to the detector (MS) connected to a computer that then collects and stores the information for each injection.

2.3 Identification of target analytes is accomplished by comparing the mass spectra of each peak with the reference spectra of authentic standards.

2.4 Quantitation of the analytes of interest is accomplished by comparing the response of a major (quantitation) ion, present in the target analyte, relative to an internal standard in each extract, in conjunction with the response factor generated from a calibration curve.

2.5 Proper quantitation ions for each compound must be selected so that no interferences are present from adjoining (or co-eluting) analytes with common ions. Proper GC conditions must be used to resolve compounds with similar mass spectra. Background subtraction of mass spectra may be necessary when matrix interference is present.

2.6 Qualitative - The identification of compounds based on retention time and comparison of the sample mass spectra, after background correction, with characteristic ions in the reference mass spectra. The reference mass spectra must be generated by the laboratory using the same analytical conditions used for the analysis of field samples. The characteristic ions from the reference mass spectra are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectra.

2.7 Quantitative – Following qualitative identification, the quantitation of the identified compound is based on the integrated abundance of the primary characteristic ion from the Extracted Ion Current Profile (EICP).

2.8 Relative Retention Time (RRT) – The process of normalizing the response (peak area) of the target compound to the response of the internal standard.

2.9 Isotope dilution calibration - Isotope dilution calibration is essentially a special case of internal standard calibration. In isotope dilution, the internal standards are stable isotopically-labeled analogs of the target analytes *and* they are added to the sample prior to any sample handling steps, including sample extraction. Because the spiked

compounds differ from the target compounds only in the presence of the stable isotopes, the physical and chemical behavior of each labeled compound is virtually the same as its unlabeled "native" analog. Thus, any losses of the target compound that may occur during any of the sample preparation, extraction, cleanup, or determinative steps will be mirrored by a similar loss of the labeled standard.

2.10 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.

2.11 Large Volume Injection (LVI): any injection volume >5µL. Technique is dependent upon type of GC inlet used and sensitivity of detection.

2.12 Minimum Level (ML): A term used in Method 625.1 which refers to either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the MDL, whichever is higher. Minimum levels may be obtained in several ways: They may be published in a method; they may be based on the lowest acceptable calibration point used by a laboratory; or they may be calculated by multiplying the MDL in a method, or the MDL determined by a laboratory, by a factor of 3. For the purposes of NPDES compliance monitoring, EPA considers the following terms to be synonymous: ‘‘quantitation limit,’’ ‘‘reporting limit,’’ and ‘‘minimum level.’’

2.13 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 The sample holding time for solid samples is 14 days to extraction and, for aqueous samples, the holding time is 7 days. Holding time begins when (date and time) the samples are collected and ends either 14 or 7 days following sampling, at the time sampled.

4.3 The holding time for each extract is 40 days from sample preparation to analysis.

4.4 The container for aqueous samples and liquid sludge being extracted using the traditional 1L EPA 3510 method are 1L amber glass bottles. For the reduced volume extraction process using the EPA 3510 method, 100mL amber glass bottles are utilized. The containers for aqueous samples being extracted using EPA Method 3511 are 40mL amber glass bottles. Add 0.008% $Na_2S_2O_3$ per liter, if residual chlorine is expected or present.

4.5 Collect solid sample materials in 4 oz. jars or larger, depending on the weight and density of the sampled materials.

4.6 All samples and extracts must be shipped and stored at <6!C (not frozen).

4.7 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

5.0 INTERFERENCES

5.1 Raw GC/MS data from all method blanks, samples, and spikes is evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample

syringe is rinsed between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of solvent to check for cross-contamination. Clean/replace injector liner or clip column, check with solvent blanks, and repeat samples if necessary.

5.3 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Attachment IV and are recommended from the published 8270 methods. There is no method stated ions for the following: Pyridine, 1-Methylnaphthalene, Biphenyl, Carbazole. Aniline Bis (2-Chloroethyl)ether and Tetraethyllead quantitation ions may vary due to chromatographic conditions causing co-elution of the shared primary ion. Targets have strongly-responding, analyte-specific secondary ions suitable for quantitative use. Refer to Attachment IV for ions.

5.4 Problematic Compounds:

5.4.1 Benzidine may be subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.

5.4.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the GC inlet, as well as photochemical decomposition.

5.4.3 N-nitrosodimethylamine may be difficult to separate from the solvent using the chromatographic conditions listed in this method.

5.4.4 N-nitrosodiphenylamine decomposes in the GC inlet and can't be separated from diphenylamine.

5.4.5 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, and Benzyl Alcohol are subject to erratic chromatographic behavior, especially when there is high boiling material contamination of the GC system.

5.4.6 Pyridine may perform poorly at the GC injection port temperatures listed in this method. The amount of degradation may be reduced by lowering the injection port temperature. Modification of the injection port temperature may adversely affect the performance of other target analytes.

5.4.7 Benzenethiol, or thiophenol, can be found in refinery wastes at caustic pH values. Benzenethiol is unstable in water/soils of neutral or acidic pH values. Benzenethiol rapidly degrades in organic solvents used to prepare the instrument calibration standards. Benzenethiol is part of Appendix VIII and the 1985 Skinner List, but was never included in Appendix IX to 40 CFR 264, due to its instability in the environment

5.4.8 Tetraethyllead may be subject to decomposition or other losses as processed using microwave digestion, 3546.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph/mass spectrometer system.

6.1.1 Gas chromatograph (HP 6890/7890 or equivalent)- An analytical system complete with a temperature- programmable gas chromatograph suitable for

split-less injection and all required accessories, including, auto sampler, syringes, analytical columns, and gases. The capillary column is directly coupled with the source.

6.1.2 Column 1 - 30m x 0.25mm ID with a 0.25µm film thickness silicon-coated fused silica capillary column (Phenomonex ZB-5MS or equivalent).

6.1.3 Column 2 – J&W 30m x 0.25mm x 0.5um film DB5MS or an equivalent is used. Ultrapure (99.999%) Helium gas is used for a mobile phase.

6.1.4 Syringes: Agilent (or equivalent) syringes sizes 10µL, 25µL, 50µL, 100µL and 1.0mL.

6.2 Mass spectrometer (HP-5973/5975 or equivalent) capable of scanning from 35 to 550 amu every 1 second, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrum for decafluorotriphenylphosphine (DFTPP) must meet the applicable criteria in method 8270C, 8270D, 8270E or 525 when 50ng of DFTPP GC/MS tuning standard is injected.

6.3 GC/MS interface - The interface is capillary-direct into the mass spectrometer source.

6.4 Data system (HP Chemstation with Enviroquant) - A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage of machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as Extracted Ion Current Profile (EICP). The most recent version of the EPA/NIST Mass Spectral Library is also available

6.5 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

6.6 Balance - Analytical, capable of weighing 0.0001g

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See SOP #030230, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

7.2 Reagent grade inorganic chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 Organic-free reagent water - all references to water in this method refer to organic-free reagent water (ASTM II or equivalent).

7.4 Burdick & Jackson Omni Solv Dichloromethane Dx0831-1 (or equivalent).

7.5 Stock standard solutions - Standard solutions are purchased as certified solutions. Commercially–prepared stock standards are used at concentrations that are certified by the manufacturer or by an independent source.

7.6 Stock standard solutions

7.6.1 Restek 8270 MegaMix – 31850, or equivalent, at 1000ppm

7.6.2 Agilent ULTRAgold Custom Standard – CUS-28559, at 1000ppm

7.6.3 NSI Lab Solutions Custom BNA Mix – Q6330, or equivalent, at 2000ppm

7.6.4 Restek Custom a-Terpineol/Quinoline Standard – 572011, or equivalent, at 2000ppm

7.6.5 Restek 8270 Benzidines Mix #2 – 31852, or equivalent, at 1000ppm

7.6.6 Restek Benzoic Acid Mix – 31879, or equivalent, at 2000ppm

7.6.7 NSI Lab Solutions 8270 BNA Mix – C-701, or equivalent, at 1000ppm

7.6.8 Phenova Custom 8270 Appendix IX Mix – ALO-130094, or equivalent, at 1000ppm

7.6.9 Phenova Benzidines Standard – ALO-101244, or equivalent, at 2000ppm

7.6.10 Phenova Benzoic Acid Mix – ALO-101246, or equivalent, at 2000ppm

7.6.11 Phenova Custom Appendix IX Mix 2 – ALO-130203, or equivalent, at 2000ppm

7.6.12 Phenova Methapyrilene – ALO-130204, or equivalent, at 2000ppm

7.6.13 Phenova Hexachlorophene – ALO-130233, or equivalent, at 4000ppm

7.6.14 Phenova Custom Appendix IX Mix 1 – ALO-130202, or equivalent, at 2000ppm

7.6.15 Phenova 8270 OP Pesticides Mix – ALO-101256, or equivalent, at 2000ppm

7.6.16 Phenova 1,4-Dioxane – ALO-101313, or equivalent, at 2000ppm

7.6.17 Restek 1,4-Dioxane – 31853, or equivalent, at 2000ppm

7.6.18 Phenova Benezenethiol – ALO-130085, or equivalent, at 1000ppm

7.6.19 Phenova Sulfolane Mix – ALO-130201, or equivalent, at 800ppm

7.6.20 Restek Sulfolane Standard – 36413, or equivalent, at 800ppm

7.6.21 Restek Custom BNA Subgroup Standard #1 – 574999, or equivalent, at 200ppm

7.6.22 Agilent ULTRAgold Custom Standard – CUS-23897, or equivalent, at 1000ppm

7.6.23 Agilent ULTRAgold Custom Standard – CUS-23952, or equivalent, at 1000ppm

7.6.24 Restek TX TPH Calibration Mix – 569373, or equivalent, at 10000ppm

7.6.25 AccuStandard Tetraethyl Lead – S-1263, or equivalent at 200ppm

7.6.26 Restek Custom N-Methylpyrrolidone Standard – 578296, or equivalent at 1000ppm

7.6.27 O2SI 1-Methyl-2-pyrrolidone Solution – 012097-01, or equivalent at 1000ppm

7.7 Transfer the stock standard solutions into bottles with PTFE-lined screw caps. Store, protected from light, at –10°C or less or as recommended by the standard manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standards are assigned a 6-month expiration date from the day that a sealed ampoule is opened. Standards are discarded if signs of degradation are apparent when compared to a second source standard.

STANDARD NAME	TOTAL VOLUME (mL)	RECIPE
BNA INTERNAL STANDARD	10	Open Ampule - No dilution required, Custom BNA Internal Standard, NSI Lab Solutions, Cat. No. Q-6343-O
1,4-DIOXANE-D8 IS @ 50ppm	10	250uL 1,4-Dioxane-d8 Standard, Restek, 30614 9.75mL Methylene Chloride
NDMA-D6 IS @ 50ppm	10	500uL 521 Surrogate Std., Restek, Cat. No. 33910 9.5mL Methylene Chloride
TUNE @ 50ppm	10	500uL GC/MS tuning std, Accu Standard, Cat. No. M-625-TS-20X 9.5mL Methylene Chloride
TUNE @ 10ppm	10	100 uL GC/MS tuning std, Accu Standard, Cat. No. M-625-TS-20X 9.9 mL Methylene Chloride
8270 PRIMARY INT @ 200ppm	10	2mL 8270 MegaMix, Restek, Cat. No. 31850 2mL ULTRAgold Custom Standard, Agilent, Cat. No. CUS-28559 6mL Methylene Chloride
8270 TCL INT @ 200ppm	10	1mL Custom BNA Mix, NSI Lab Solutions, Cat. No. Q6330 1mL Custom a-Terpineol/Quinoline Standard, Restek, Cat. No. 572011 1mL 8270 Benzidines Mix #2, Restek, Cat. No. 31852 1mL Benzoic Acid Mix, Restek, Cat. No. 31879 6mL Methylene Chloride
8270 SVMS SSCV @ 10ppm	1	10uL 8270 BNA Mix, NSI Lab Solutions, Cat. No. C-701 10uL ULTRAgold Custom Standard, Agilent, CUS-28559 980uL Methylene Chloride
8270 TCL SSCV @ 200ppm	10	2mL Custom 8270 Appendix IX Mix, Phenova, Cat. No. ALO-130094 1mL Benzidines Standard, Phenova, ALO-101244 1mL Benzoic Acid Mix, Phenova, ALO-101246 1mL Custom Appendix IX Mix 2, Phenova, Cat. No. ALO-130203 5mL Methylene Chloride
8270 TCL SSCV @ 10ppm	1	100uL 8270 TCL SSCV @ 200ppm, 900uL Methylene Chloride
SVAM INT @ 200ppm	10	1mL Custom Appendix IX Mix 2, Phenova, ALO-130203 1mL Methapyrilene, Phenova, Cat. No. ALO-130204 1mL Hexachlorophene, Phenova, Cat. No. ALO-130233 7mL Methylene Chloride
SVAP INT @ 200ppm	10	1mL Custom Appendix IX Mix 1, Phenova, Cat. No. ALO-130202 9mL Methylene Chloride
SVOP INT @ 200ppm	10	1mL 8270 OP Pesticides Mix, Phenova, Cat. No. ALO-101256 9mL Methylene Chloride

NDMA/1,4-DIOXANE INT @ 20ppm	10	100uL 1,4-Dioxane, Phenova, Cat. No. ALO-101313 1mL 8270 Primary Int. @ 200ppm, 8.9mL Methylene Chloride
NDMA/1,4-DIOXANE INT @ 2ppm	5	500ul NDMA/1,4-Dioxane Int @ 20 4.5mL Methylene Chloride
1,4-DIOXANE SSCV @ 200ppm	10	1mL 1,4-Dioxane, Restek, Cat. No. 31853 9mL Methylene Chloride
1,4-DIOXANE SSCV @ 10ppm	10	500uL 1,4-Dioxane SSCV @ 200pm 9.5mL Methylene Chloride
NDMA SSCV @ 10ppm	10	100uL 8270 BNA Mix, NSI Lab Solutions, Cat. No. C-701 9.9mL Methylene Chloride
BENZENETHIOL CAL INT @ 100ppm	10	1mL Benzenethiol, Phenova, Cat. No. ALO-130085 1mL Ultragold Custom Standard, Agilent, CUS-28559 8mL Methylene Chloride
BENZENETHIOL SSCV @ 100ppm	10	1mL Benzenethiol, Phenova, Cat. No. ALO-130085 9mL Methylene Chloride
SULFOLANE CAL INT @ 50ppm	10	625uL Sulfolane Mix, Phenova, Cat. No. ALO-130201 500uL Ultragold Custom Standard, Agilent, CUS-28559 8.875mL Methylene Chloride
SULFOLANE SSCV @ 50ppm	10	625uL Sulfolane, Restek, Cat. No. 36413 9.375mL Methylene Chloride

7.8 Internal standards solutions- the internal standards are naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, perylene-d12 and 1-4 dichlorobenzene-d4. Purchase from NSI (Cat # Q-6343-O) as certified stock solution at 800µg/mL. Alternative internal standard concentrations may be used for LVI work. Internal standard intermediates at 16µg/mL and 4µg/mL are prepared for spiking, RV/LVI 8270PAHand RV/LVI 8270SIM analyses, respectively.

7.8.1 For all concentrated soil and 8270 full run water reduced volume extracts, use the 8000µg/mL internal standard solution. Each sample extract undergoing analysis is spiked with 5µL of internal standard intermediate solution, resulting in a concentration of 8µg/mL of each internal standard.

7.8.2 For non-concentrated soil, reduced volume water and 3511 water analyses, including PAH and DROMO, use the 16µg/mL ISTD intermediate. For non-concentrated soil, reduced volume and EPA 3511 water extracts being analyzed by the SIM process, use the 4µg/mL ISTD intermediate. Each sample extract undergoing analysis is spiked with 10µL of the appropriate internal standard intermediate solution, resulting in a concentration of 160µg/L and 40µg/L, respectively, for each internal standard.

7.9 Using a volumetric syringe, measure each of the solutions listed in Section 7.9 and place into a 10mL volumetric flask.

7.10 Preparation of Working Standards

Standards must be stored at ≤6!C. The expiration date of any working standard will be 6 months unless the manufacturer's stock expires prior to that date or if the standard starts showing signs of degradation. See section 7.11.1 through 7.11.6 for preparation instructions. Concentrations of standards used are subject to change depending on instrument condition, client needs and sample preparation method of the variety of

analysis being performed. A minimum of five calibration levels is required for Method 8270C and 8270D, while a minimum of 3 calibration levels is required for Method 625.

7.10.1 8270C/D/E Calibration standards for concentrated soil and 1L concentrated water extractions: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory's reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediate stock standard described in section 7.10 give solutions at concentrations of 0.2µg/mL up to 50µg/mL. Surrogates are included at the same concentrations. Internal standards are spiked at a constant concentration per extraction method for quantitation purposes.

SVOC mix (200ppm) µL	ISTD mix uL	Final volume	Final conc. ppm	Level
1	10	1.0mL	0.2	1
5	10	1.0mL	1	2
10	10	1.0mL	2	3
25	10	1.0mL	5	4
50	10	1.0mL	10	5
75	10	1.0mL	15	6
100	10	1.0mL	20	7
150	10	1.0mL	30	8
200	10	1.0mL	40	9
250	10	1.0mL	50	10

A minimum of 5 points are used to construct the calibration curve.

7.10.2 Calibration standards for 8270C/D/E reduced volume and EPA 3511 (soil and water) extracted samples: A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system. Each standard contains each analyte for detection. Working standards are made directly from the intermediates described in section 7.9 to give solutions at concentrations of 0.01µg/mL up to 1µg/mL. Surrogates are included at the same concentrations. Internal standards are spiked at a constant of 160µg/L for quantitation purposes.

SVOC mix (2ppm) µL	ISTD mix uL	Final volume	Final conc. ppb	Level
5	10	1.0mL	10	1
25	10	1.0mL	50	2
50	10	1.0mL	100	3
100	10	1.0mL	200	4
200	10	1.0mL	400	5
300	10	1.0mL	600	6
400	10	1.0mL	800	7
500	10	1.0mL	1000	8

7.10.3 For SIM analyses concentrated soil and 1L water extractions, calibration standards are diluted from the intermediate standard solution (section 7.9.1) to give a calibration at the following concentrations: 20, 50, 100, 500, 1000, 2000, 4000, 10,000µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 8µg/mL.

SIM Standard Concentration (ug/L)	Amount Added (uL) 5µg/mL Int.	Final Volume (mL)
20	2.0	1.0
50	5.0	1.0
100	10.0	1.0
500	50.0	1.0
1000	100.0	1.0
2000	200.0	1.0
4000	400.0	1.0
10000	1000.0	1.0

7.10.4 For SIM analyses using reduced volume, non-concentrated soil, or EPA 3511 extracts, calibration standards are diluted from the intermediate standard solution (section 7.9.3) to give a calibration at the following concentrations: 1, 5, 10, 20, 40, 80, 200µg/L. A minimum of five calibration standards is prepared at different concentrations. At least one of the calibration standards must correspond to a sample concentration at or below the laboratory-reporting limit (RL). The calibration levels may change based on the working range of the GC/MS system. Surrogates are included at the same concentrations. The internal standards are at a constant 40µg/L.

SIM RV 3511 Standard Concentration (ug/L)	Amount Added (uL) 200µg/L Int.	Final Volume (mL)
1	5	1.0
5	25	1.0
10	50	1.0
20	100	1.0
40	200	1.0
80	400	1.0
200	1000	1.0

7.10.5 For Missouri DRO analysis by 3511 and non-concentrated soil, prepare the working calibration curve as reflected in the following table.

DROMO mix (200ppb) µL	Final Volume	Final conc. (ppb)
25	1.0 mL	5
50	1.0 mL	10
100	1.0 mL	20
200	1.0 mL	40
400	1.0 mL	80
600	1.0 mL	120
800	1.0 mL	160

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STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, D, E & 625.1) Including Provisions for Analysis in SIM Mode
ISSUER: Pace National – Mt. Juliet, Tennessee

8.0 PROCEDURE

STATE NOTE: For samples analyzed in conjunction with the Ohio VAP program, the criteria found and itemized in this procedure for EPA method 8270C must be utilized. Alternative GCMS tuning criteria from that specified in EPA 8270C is acceptable as permitted in Section 7 of the published method.

8.1 GC Conditions: The GC conditions are listed in each instrument maintenance log and are updated as necessary.

8.1.1 Due to the tuning and calibration requirements outlined in this method, a liner change is necessary prior to beginning an analytical sequence. Any additional maintenance performed on the instrumentation will be documented as performed in the specific instrument maintenance log. (i.e., column clip/change, septa change, inlet cleaning, detector cleaning/maintenance, etc.)

8.2 Mass Spectrometer Tuning Criteria: The GC/MS is hardware-tuned using a 50ng (or less) injection of DFTPP. Analyses must not begin until the tuning criteria are met. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex & ±1 scan) and average the three scans then perform background subtraction. If Autofind is not utilized, select the mass spectrum at the peak apex for evaluation, or use an average mass spectrum across the entire DFTPP peak. Background subtraction is required prior to the start of the peak but no more than 20 scans prior. Background correction cannot include any parts of the target peak. The scans must be averaged and background corrected.

The mass spectrometer must be tuned every 12 hours if samples, standards, etc. are to be analyzed for Methods 8270C, 8270D and 625.1. The mass spectrometer must be tuned prior to initial calibration only when samples, standards, etc. are to be analyzed for Method 8270E. Pace National uses 8270D evaluation criteria per method allowances.

TABLE 8.2
Method 8270D
DFTPP Key Ions And Ion Abundance Criteria[a, b]

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present, but <24% of mass 442
442	Base peak, or >50% of mass 198
443	15-24% of mass 442

(a) Data taken from Table 3 in SW-846 Method 8270D.
(b) Alternate tuning criteria may be used (e.g., CLP, Method 525, or manufacturers' instructions), providing that method performance is not adversely affected.

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STATE NOTE: All South Carolina samples require a tune every 12 hours, regardless of which method is being utilized.

The GC/MS tuning standard solution must also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD is used to assess breakdown occurring in the injection port. The calculation for the determination of the breakdown occurring is found in section 9.1 and must include both DDD and DDE. Breakdown must not exceed 20%. Benzidine and pentachlorophenol total ion chromatogramsl are used to assess tailing occurring within the analytical system and both analytes should be present at their normal responses with no obvious peak tailing. To determine the tailing factor for benzidine and pentachlorophenol, use the calculation found in section 9.2. For EPA Methods 625 and 8270C, benzidine must have a tailing ratio of <3 and pentachlorophenol must have a tailing ratio of <5. For EPA Method 8270D and 625.1, benzidine and pentachlorophenol must have a tailing ratio of <2. The Missouri diesel method does not require tailing or degradation checks prior to or during analysis.

8.3 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below: Additional guidance for performing SIM analyses, in particular for PAHs and phenol target analyte compounds, can be found in the most recent CLP semivolatile organic methods statement of work (SOW). See the SIM sections from the following CLP SOW for further details: EPA CLP Organics SOW. (Reference 14)

SIM Groups for PAHs and including pentachlorophenol, hexachlorobenzene and tetraethyllead

SIM Group	1	2	3	4	5	6	7	8
RT start	Solvent delay	Before 2-Methyl naphthalene	Before Acenaph-thalene	Before Fluorene	Before Fluor-anthene	Before Benzo (a)-anthracene	Before Benzo (b)-fluoranthene	Before Dibenz (a,h-)anthracene
Ions	82, 128, 129, 136, 137, 237, 295	127, 141, 142, 162, 171, 172	139, 151, 152, 153, 154, 162, 164, 168	94, 165, 166, 176, 178, 179, 188, 264, 266, 268, 282, 284, 286	200, 202, 203, 244, 245	226, 228, 229, 240, 241	252, 253, 260, 264	138, 139, 276, 277, 278, 279
Dwell	40	35	25	30	40	50	75	50

8.4 Calibration

8.4.1 **Initial Calibration**

EPA Method 8270C: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see Section 13.4). See section 8.3.2 for information regarding use and deletion of calibration points.

The calibration check compounds (CCCs) listed in Section 8.3.1a must have an average percent relative standard deviation (%RSD) of less than or equal to 30%. Any target analyte that has a %RSD >15% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤15%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or quadratic curve fit. The calibration curve cannot be forced through zero and does not include a method blank. It must also meet a correlation coefficient of 0.995 or better. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve.

The system performance check compounds (SPCCs) in Table 8.3.1b must have an average RF of ≥0.05. When these criteria are met, samples can be analyzed.

Table 8.3.1a: Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
n-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Table 8.3.1b: System Performance Check Compounds (SPCC)

Compound	*Minimum Average Response Factor*
n-Nitroso-di-n-propylamine	>0.05
Hexachlorocyclopentadiene	>0.05
2,4-Dinitrophenol	>0.05
4-Nitrophenol	>0.05

EPA Method 8270D/E: The working standards prepared in section 7.10 are injected and average response factors are calculated. The calibration curve is typically constructed of six to nine standards, however, this may change depending on instrument conditions and/or client needs (see section 13.4). At least five standards are required for Response Factor and linear regression calibration. If a quadratic curve fit is used, a minimum of 6 calibration standards must be utilized to obtain a working calibration curve. See section 8.3.2 for

information regarding use and deletion of calibration points.

Target analytes must have an average RSD of ≤20%. Any target analyte that has a %RSD >20% for the RF must be calculated by linear or quadratic regression instead of RF. If the RSD of any target analyte is ≤20%, the average response factor may be used for quantitation. When any compound does not meet the calibration criteria for RF, the analyst MUST use linear regression or, if permitted, quadratic curve fit. The calibration curve cannot be forced through zero. It must also meet a correlation coefficient of 0.995 or better.

8270D RF evaluation: In addition to the minimum %RSD criteria, it is recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level to ensure that these compounds are performing as expected. See Table 8.3.1c. Meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

8270E RF evaluation: In addition to the minimum %RSD criteria, it is recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level to ensure that these compounds are performing as expected. See Table 8.3.1c. Because the minimum RFs in Table 8.3.1c were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information is provided as guidance only. The laboratory should establish procedures in its SOP (e.g., laboratory established minimum RFs, signal-to-noise (S/N) checks, etc.) to ensure that the instrument is working properly and that calibration standards were correctly prepared.

Table 8.3.1c: Recommended Minimum Response Factors for Each Calibration Level (Initial and Continuing Calibration)

Compound	*Minimum Response Factor*	*Compound*	*Minimum Response Factor*
Benaldehyde	0.010	Bis(2-chloroethoxy)methane	0.300
Phenol	0.800	2,4-Dichlorophenol	0.200
Bis(2-chloroethyl)ether	0.700	Naphthalene	0.700
2-Chlorophenol	0.800	4-Chloroaniline	0.010
2-Methylphenol	0.700	Hexachlorobutadiene	0.010
2,2-Oxybis-(1-chloropropane)	0.010	Caprolactam	0.010
Acetophenone	0.010	4-Chloro-3-methylphenol	0.200
4-Methylphenol	0.600	2-Methylnaphthalene	0.400
n-Nitroso-di-n-propylamine	0.500	Hexachlorocyclopentadiene	0.050
Hexachloroethane	0.300	2,4,6-Trichlorophenol	0.200
Nitrobenzene	0.200	2,4,5-Trichlorophenol	0.200
Isophorone	0.400	1,1-Biphenyl	0.010
2-Nitrophenol	0.100	2-Chloronaphthalene	0.800
2,4-Dimethylphenol	0.200	2-Nitroaniline	0.010

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Compound	*Minimum Response Factor*	*Compound*	*Minimum Response Factor*
Dimethyl phthalate	0.010	Phenanthrene	0.700
2,6-Dinitrotoluene	0.200	Anthracene	0.700
Acenaphthylene	0.900	Carbazole	0.010
3-Nitroaniline	0.010	Di-n-butyl phthalate	0.010
Acenaphthene	0.900	Fluoranthene	0.600
2,4-Dinitrophenol	0.010	Pyrene	0.600
4-Nitrophenol	0.010	Butyl Benzyl phthalate	0.010
Dibenzofuran	0.800	3,3-Dichlorobenzidine	0.010
2,4-Dinitrotoluene	0.200	Benzo(a)anthracene	0.800
Diethyl phthalate	0.010	Chrysene	0.700
1,2,4,5-Tetrachlorobenzene	0.010	Bis (2-ethylhexyl)phthalate	0.010
4-Chlorophenyl-phenyl ether	0.400	Di-n-octyl phthalate	0.010
Fluorene	0.900	Benzo(b)fluoranthene	0.700
4-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
4,6-Dinitro-2-methylphenol	0.010	Benzo(a)pyrene	0.700
4-Bromophenyl-phenyl ether	0.100	Indeno(1,23-c,d)pyrene	0.500
n-Nitrosodiphenylamine	0.010	Dibenz(a,h)anthracene	0.400
Hexachlorobenzene	0.100	Benzo(g,h,i)perylene	0.500
Atrazine	0.010	2,3,4,6-Tetrachlorophenol	0.010
Pentachlorophenol	0.050		

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 8270C SIM, all target compounds must be treated as CCCs and must have an average RSD of ≤30%.

EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.1 (EPA Method 8270D).

EPA 8270E GC/MS SIM: If analyzing samples by EPA 8270E SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.1 (EPA Method 8270E).

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EPA Method 625.1: One of the calibration standards should be at a concentration at or below the reporting limit. The resulting calibration must meet all applicable acceptance criteria in Section 10, based on the RSD, RSE, or r^2. The concentrations of the other calibration standards should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system for full-scan and/ or SIM operation, as appropriate. A minimum of six concentration levels is required for a second order, non-linear (i.e., quadratic) calibration.

Calculate the mean (average) and relative standard deviation (RSD) of the responses factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios, As/Ais, vs. concentration ratios Cs/Cis. If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (r^2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error (RSE) may be used as an

acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated.

All Published Methods: Reference spectra must be updated upon analysis of each new calibration curve.

Linear Regression Weighting: As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear or second order regression calibration curve, by plotting A/A(is) vs. Q(x) using the equations found in section 9.4. Either equal weighting factors or 1/x regressions may be used.

STATE NOTE: For all Minnesota sample analyses, the RL level standard is re-injected and quantitated against the newly updated calibration curve or the applicable standards are reprocessed (re-quantitated) using the completed calibration curve and is evaluated for the ±40% deviation criterion with the exception of the listed poor performers in this procedure.

STATE NOTE: For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40% RSD is used for normal target analytes and 50% RSD is utilized for known poor performing compounds.

STATE NOTE: When analyzing samples in conjunction with the Ohio VAP or South Carolina DHEC programs, the calibration model must be RSD or linear. Quadratic curve modeling is not permitted unless historical performance of analytes exhibited a nonlinear response (i.e., Benzoic Acid and problematic phenols). Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance.

8.4.2 CALIBRATION POINTS – Usage and Deletion

When the appropriate number of calibration standards is used, all points must be considered in the average response factor calculation or linear regression calculation. The deletion of the highest point is acceptable when necessary, with the analyst noting that the high end of the calibration has been lowered. The deletion of the lowest calibration point is acceptable, when necessary, provided that the analyst notes the deletion on the injection log and raises the reporting limit, if necessary, for that compound.

8.4.3 **EPA Method 8270D/E:** LINEAR REGRESSION USE – The method of linear regression calibration has the potential for a significant bias to the lower portion of the calibration model. This bias is not normally seen in relative percent difference methods. When utilizing linear regression fits, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the lowest concentration standard back into the completed calibration curve. It is not necessary to re-analyze a low concentration standard, but using the analytical system software, the low standard can be re-

quantitated as if it were a field sample. The recalculated concentrations of the analytes utilizing the linear regression curve fit must be within ±50% of the true standard concentration.

STATE NOTE: For the analysis of South Carolina samples, all target analytes, including Hexachlorophene, is required to utilize linear regression. Quadratic curve fit is not allowed. To achieve this, the calibration curve may be modified by the removal of the lowest two levels and will utilize calibration levels of 60, 80, 100, 120, and 140 for quantitation of this analyte in South Carolina samples. The reporting limit (RL) for South Carolina will routinely be 100ppb for water samples.

8.4.4 Quadratic Regression: Quadratic regression may be used for the following compounds: Pentachlorophenol, 4-Nitrophenol, 2,3,4,6-Tetrachlorophenol, 4,6-Dinitro-2-methylphenol, 2,4-Dinitrophenol. Plots must have a minimum of 6 points and a correlation coefficient of 0.995 or better.

STATE NOTE: Quadratic curve modeling is not permitted for samples originating in South Carolina or for samples reported for the Ohio VAP unless historical performance of analytes exhibited a nonlinear response (e.g., Benzoic Acid).

8.4.5 **Acceptance criteria independent of calibration model –** Either of the two procedures described in Secs. 8.4.5.1 and 8.4.5.2 may be used to determine calibration function acceptability for linear and non-linear curves. These include refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

8.4.5.1 Calculation of the % Error (see section 9.5 for calculation) Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

8.4.5.2 Calculation of Relative Standard Error (See section 9.6 for calculation) The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for average CF or average RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds. A list of known poorly performing compounds can be found in Sec. 16 of this document.

8.4.6 Second Source Calibration Verification – the initial calibration for each target analyte must be checked with a standard from a source that is different from those used for initial calibration.

8.4.7 **Daily Tuning and Continuing Calibration**

8270E: Daily analysis of the GC/MS tune check solution is not required as part of the CCV. The analyst should, however, closely monitor

chromatography as well as target and IS responses in the CCV for deterioration in the system.

8270C/D, 625.1: As with the initial calibration, the system must be tuned with 50ng of DFTPP or less to meet the acceptance criteria found in section 8.1. Following successful tuning, the midpoint level standard (CCV) is analyzed. Calibration verification for each method, as listed below, must be met prior to the analysis of field samples and every 12 hours for 8270C/D and every 24 hours for EPA 625 (see the method note in Section 8.2 for Method 625.1 requirements).

EPA Method 8270C: The percent difference of the CCCs (see Table 8.3.1a & b) in the mid-level standard must be $\leq$20% and the SPCCs must have an RF $\geq$0.05. The retention time of the internal standards must be within $\pm$30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within –50% to +100%. Once these criteria are met, samples can be analyzed.

EPA Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration. Include the surrogates in this solution. It is necessary to verify calibration for the analytes of interest only.

Compare the recoveries for the analytes of interest against the acceptance criteria for recovery (Q) in Attachment VII and the recoveries for surrogates against the acceptance criteria in Attachment VIII. If recovery of the analytes of interest and surrogates meet acceptance criteria, system performance is acceptable and analysis of samples may continue. If any individual recovery is outside its limit, system performance is unacceptable for that analyte.

EPA Methods 8270C and 625 (analyzed concurrently): The CCV must be evaluated for CCC and SPCCs as per EPA Method 8270C requirements. All non-CCC and other target analytes must meet the criteria established in Method 625 for all analytes ($\pm$20%). For analytes not contained in the Method 625 analyte list, the analyst evaluates the CCV and the experience of the analyst weighs heavily in determining the usability of the data.

STATE NOTE: For all Wisconsin sample analyses, non-CCC compounds for 8270C requires a $\pm$50% criteria for the CCV.

EPA 8270C GC/MS SIM: When analyzing samples using SW-846 EPA 8270C SIM, all compounds in the CCV must be treated as CCCs and must meet the minimum requirements of $\leq$20% difference.

EPA 8270D GC/MS SIM: If analyzing samples by EPA 8270D SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.6 (EPA Method 8270D below).

EPA 8270E GC/MS SIM: If analyzing samples by EPA 8270E SIM, follow the initial calibration criteria for the specified referenced method as found in section 8.3.6 (EPA Method 8270E below).

EPA Method 8270D: Each of the most common target analytes in the CCV must meet the minimum response factors in Table 8.3.1c. When using the average RF, the percent difference for each target compound in the CCV must be ≤20%. When using regression fit calibration, the percent drift of the CCV must be ≤20%. The retention time of the internal standards must be within ±30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within –50% to +100%.

EPA Method 8270E: Each of the most common target analytes in the CCV may but is not required to meet the minimum response factors in Table 8.3.1c. When using the average RF, the percent difference for each target compound in the CCV must be ≤20%. When using regression fit calibration, the percent drift of the CCV must be ≤20%. The retention time of the internal standards must be within ±30 seconds from the mid-point standard level of the last initial calibration curve and the area response must be within –50% to +100%.

8.4.8 For corrective action regarding tuning and calibration, see sections 11.1 and 11.2.

8.5 Method Blank Analysis – A method blank should be analyzed prior to any field sample analysis to verify that the analytical system is free from contaminants. If the method blank indicates that contamination may be present in the analytical system, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts.

8.6 GC/MS analysis of field samples and preparation QC.

8.6.1 It is highly recommended that the extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

8.6.2 Allow the extracts to warm to room temperature. Just prior to analysis, add 10µL of the internal standard solution to the 1mL concentrated extract or 5µL of the internal standard solution to the 0.5mL extract obtained from sample preparation.

8.6.3 If the response for any quantitation ion exceeds highest level of the initial calibration range, the extract must be diluted and re-analyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (0.04, 0.16 or 8ng/uL, unless a more sensitive GC/MS system is being used). For example, if performing a 1:10 dilution on a concentrated extract, take 100uL of the extract and dilute to a volume of 1mL with the appropriate solvent. Add 9uL of the appropriate internal standard solution to the diluted extract and inject on the analytical system. It can be assumed that 1uL of internal standard was contained in the 100uL extract used for the initial dilution.

8.6.4 Internal standard area counts and retention times must be monitored in all samples, spikes and method blanks to monitor system performance, check for drifting, ensure effective autosampler performance, etc. If the area of the Extracted Ion Current Profile (EICP) changes by a factor of 2 (-50% to +100%) from the areas in the daily CCV, corrective action is required. The RRT of the internal standard in the extract must be within ± 0.06RRT units of the RRT of the daily CCV.

STATE NOTE: With each new calibration curve, a reporting limit verification (RLV) standard must be analyzed for samples analyzed from Minnesota. This standard consists of either re-injecting the low calibration standard(s) or re-processing the low standard(s) utilized in the construction of the calibration curve. The RLV must recover within ±40% of the expected concentration. See section 11.10 for additional information.

8.7 Qualitative Identification

8.7.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Retention time windows for internal standards and target compounds integrations are updated with each calibration curve and after any instrument maintenance occurs that causes a shift that may affect ChemStation integrations.

8.7.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

8.7.1.2 The RRT of the sample component is within ± 0.06RRT units of the RRT of the standard component.

8.7.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

(EXAMPLE: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Analyst experience is vital in this determination when interferences are present.

8.7.1.4 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 the two isomers is <50% of the average of the two peak heights (for Method 8270D/E) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers are identified as isomeric pairs.

8.7.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

8.7.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

8.7.1.7 Absolute retention times are used for compound identification in all GC methods that do *not* employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that may not be confirmed.

8.7.1.7.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all standard mixtures over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

8.7.1.7.2 Record the retention time (in minutes) for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.

8.7.1.7.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

8.7.1.7.4 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

8.7.1.7.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples

run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

8.7.1.7.6 Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed or if a GC column has been shortened during maintenance.

8.8 TICs – Tentatively Identified Compounds

Periodically, clients may request the tentative identification of compounds present in the field sample that are not normal target compounds and are not normally calibrated. This identification is limited to the compounds in the current NBS (National Bureau of Standards) mass spectral library employed by Pace National.

Library Search Identification – For samples containing components not associated with the calibration standards, a library search may be made for the purpose of a tentative identification. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when making comparisons. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. The analyst may only assign tentative identifications after visual comparison of sample spectra with the nearest library searches.

Guidelines for tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >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% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- 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.

Routinely, Pace National employs a minimum Q value of 80 for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 80 but above 10ppb are reported as “Unknown”. Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (I.e., siloxanes) are also removed.

8.9 Quantitative analysis

8.9.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

8.9.1.1 It is recommended to use the integrations produced by the software if the integration is correct because the software will produce more consistent integrations of peaks in chromatograms. Manual integrations may be necessary in some cases and must be performed in conjunction with ENV-SOP-CORQ-0006, *Manual Integration*.

DOD samples must include a reason for each integration performed on the manual integration documentation.

8.9.2 If the RSD of a compound's response factor meets method requirements, then the concentration in the extract may be determined using the average response factor (average RF) from initial calibration data.

8.9.3 Where applicable, the concentration of any tentatively identified compounds in the sample should be estimated. The same formula as is used to calculate target analyte concentrations is used with the following modifications: The areas A_x and A_{is} must be from the total ion chromatograms and the RF for the compound is assumed at 1. See section 9.7 for calculation.

8.9.4 The resulting concentration must be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences for estimated concentration calculations.

8.9.5 Quantitation of multi-component compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, by Methods 8081or 8082. However, Method 8270 may be used to confirm the identification of these compounds, when the concentrations are at least 10ng/µL in the concentrated sample extract.

8.9.6 **Peak Resolution:** Structural isomers that produce very similar spectra must be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two isomers is <50% of the average of the two peak heights (for Method 8270D/E) and <25% of the sum of the two peak heights (for Methods 8270C & 625). Otherwise, structural isomers should be identified as isomeric pairs.

STATE NOTE: Minnesota MPCA requires that peak resolution of all co-eluters, analyzed using Method 8270C, must be resolved as close to 75% as possible. Resolution must be adequate at lower levels and not worsen as concentration increases.

8.9.7 Indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene share a similar structure and physical properties. Under routine analytical production conditions it is very difficult to achieve resolved chromatographic separation. The mass-spectra of these compounds exhibit base peaks separated by 2 AMUs (276 and 278 respectively) and these unique ions are used for quantitation of the respective compounds as defined by Method 8270. It has been found that the major base ion, 276, for indeno(1,2,3-cd)pyrene includes a significant contribution from dibenz(a,h)anthracene when the targets are present together at equal concentrations; however, indeno(1,2,3-cd)pyrene presence *does not* contribute significant ion 278 abundance to dibenz(a,h)anthracene quantitation at equal concentrations. For these reasons when dibenz(a,h)anthracene is found to be present at similar or lesser concentrations than indeno(l,2,3-cd)pyrene, the results are normalized by the calibration conditions and considered to be non-

impacted. Alternatively, when dibenz(a,h)anthracene is found to be present at relatively greater concentrations than indeno(l ,2,3-cd)pyrene, the indeno(1,2,3-cd)pyrene results are considered to be elevated and may be confirmed by a secondary acquisition and analysis utilizing a technique for chromatographic separation of the targets. Concentrations shall be considered similar up to a factor of two.

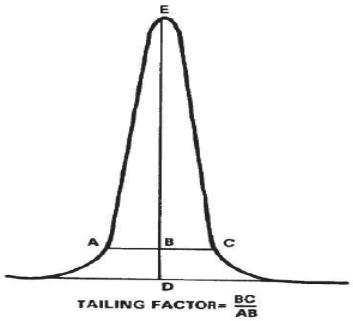
9.0 DATA ANALYSIS AND CALCULATIONS

9.1 GC/MS Tune: DDT Breakdown Determination during Tuning:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

9.2 GC/MS Tune: Benzidine and Pentachlorophenol Tailing Factor

$$\text{TailingFactor} = \frac{BC}{AB}$$



where: BC is the width of the back ½ of the peak at 10% of the peak height
AB is the width of the front ½ of the peak at 10% of the peak height.

9.3 Internal Calibration Equations (Response Factors):

$$RF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

- Percent Relative Standard Deviation (%RSD)

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100\%$$

where:
RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

- Concentration of an analyte in an extract using RF (on column):

$$X_s = \frac{(Conc._{ISTd})(Area_{Analyte})}{(Average\ RF_{analyte})(Area_{ISTd})}$$

where:
X_s = Calculated raw concentration of analyte (in ppb)

- Quantitation Report Multiplier"

$$M_a = \frac{(V_t)(D)}{(V_s)} \quad or \quad M_s = \frac{(V_t)(D)}{(W_s)}$$

where:
M_a = Quantitation Report Multiplier for Aqueous Samples
M_s = Quantitation Report Multiplier for Solid Samples
V_t = Total volume of concentrated extract (in mL)
D = Dilution factor. If no dilution, D=1. Always dimensionless
V_s = Volume of aqueous sample extracted (in mL)
W_s = Weight sample extracted (in grams)

- Sample concentration by volume (ug/L) for aqueous samples:

$$Concentration\ in\ \frac{\mu g}{L} = (X_s)(M_a)$$

- Sample concentration by weight (ug/kg) for solid samples and non-aqueous liquids:

$$Concentration\ in\ \frac{\mu g}{kg} = \frac{(X_s)(M_s)}{(\%S)}$$

where:
%S = Percent solids expressed as a decimal

9.4 Relative Retention Time (RRT):

$$RRT = \frac{RT\ of\ Target\ Analyte}{RT\ of\ Internal\ Standard}$$

9.5 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} + 100$$

where:
x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units
x_i = True amount of analyte at calibration level *i*, in mass or concentration units

9.6 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 625.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2}{(n-p)}$$

where:
x'i = Calculated concentration at level i
xi = Actual concentration of the calibration level i
n = Number of calibration points
p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

9.7 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Method 625.1 Demonstration of Capability (DOC) Requirements

10.1.1.1 For the DOC, a QC check sample (LCS) concentrate containing each analyte of interest is prepared in a water miscible solvent. The QC check sample concentrate must be prepared independently from those used for calibration, but may be from the same source as the second-source standard used for calibration verification. The concentrate should produce concentrations of the analytes of interest in water at the midpoint of the calibration range, and may be at the same concentration as the LCS.

10.1.1.2 Prepare four QC check samples and a blank sample by adding an appropriate volume of the concentrate and of the surrogate(s) to each of four aliquots of reagent water and mix well. For the blank sample, add the appropriate amount of surrogate to the reagent water and mix well. The volume of reagent water must be the same as the volume that will be used for the sample, blank, and MS/MSD.

10.1.1.3 Extract and analyze the four LCSs samples.

10.1.1.4 Calculate the average percent recovery ($\overline{X}$) and the standard deviation (s) of the percent recovery for each analyte using the four results.

10.1.1.5 For each analyte, compare s and $\overline{X}$ with the acceptance criteria for precision and recovery presented in Attachment VII. For analytes that are not listed, QC acceptance criteria must be developed by the laboratory.

If s and $\overline{X}$ for all analytes of interest meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples can begin. If any individual s exceeds the precision limit or any individual $\overline{X}$ falls outside the range for recovery, system performance is unacceptable for that analyte.

10.1.1.6 When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Batches:

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Matrix Spike/Spike Duplicate (MS/MSD) (if client has supplied sufficient sample volume). All batch information must be maintained in the preparation documentation assigned to the department.

10.4 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 1.8.2) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels

10.4.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).

10.4.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.

10.4.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within 3 years.

10.4.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.

10.5 For acceptance criteria for calibration standards, QC samples and field samples and corrective actions, see section 11.0.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 A successful DFTPP tune must be achieved prior to initial calibration or daily calibration verification(daily tuning prior to CCV not required for 8270E). If a tune does not meet the acceptance criteria in section 8.2, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called “Autotunes” that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.Following successful tuning of the DFTPP solution, the DDT degradation and Benzidine/Pentachlorophenol tailing must be assessed using the total ion chromatograms for each. If either fail to meet the required acceptance criteria, instrument maintenance is required. The DDT degradation is most likely an inlet or column condition and corrective action entails clipping 6-12” from the injector end of the column, changing the injection port liner, possibly changing the gold inlet seal and re-injecting the tuning solution. The tailing issue is most likely caused by the same type of inlet issues and the same corrective action steps should occur when the tailing criteria is not met. Tailing may also be caused by incorrect column positioning in the inlet and the correct position of the column should be verified prior to performing more involved corrective action processes.

A successful instrument tune, including degradation and tailing acceptability, must be achieved prior to the analysis of calibration standards and sample extracts by methods 8270C/D and 625.1. 8270E does not require tuning at any time other than prior to initial calibration

11.2 Initial or Continuing Calibration:

Method 8270C, SM 6410B & Method 625.1: If the calibration curve or daily calibration verification fails to meet the applicable method verification criteria for RSD, the analyst MUST use linear regression or quadratic curve fit. Quadratic models cannot be used to extend the calibration range or bypass instrument maintenance. If the method criteria are still not met when using the alternate curve fits, samples may not be quantitated using the calibration curve and a new calibration curve must be analyzed. Instrument maintenance and/or new standard preparation may also be required prior to the analysis of the new calibration curve. Following maintenance, the new calibration curve can be generated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the

manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

Method 8270D/E: Due to the large number of compounds that may be analyzed by this method, some compounds in the initial and/or daily calibration verification will fail to meet the initial and continuing calibration acceptance criteria. For these instances, failing compounds may not be critical to specific project needs and therefore may be utilized as qualified data or estimated values for screening purposes. If more than 10% of the compounds in the initial or continuing calibration exceed the 20% RSD limit and/or do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis. Instrument maintenance must be performed and the calibration process must be repeated. The system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer. Additional actions that can be taken to address failures in calibration are included in section 8.3.

TNI: If the ICV or CCV results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 9.5). Percent error between the calculated and expected amounts of an analyte must be ≤30% for all standards. (≤50% is acceptable for the lowest calibration point.)

Method 625.1: The RF or calibration curve must be verified immediately after calibration and at the beginning of each 12-hour shift, by analysis of a standard at or near the concentration of the mid-point calibration standard. The standard(s) must be obtained from a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration.

When one or more analytes fail acceptance criteria, analyze a second aliquot of the calibration verification standard and compare ONLY those analytes that failed the first test with their respective acceptance criteria. If these analytes now pass, system performance is acceptable and analysis of samples may continue. A repeat failure of any analyte that failed the first test, however, will confirm a general problem with the measurement system. If this occurs, repair the system and repeat the test, or prepare a fresh calibration standard and repeat the test. If calibration cannot be verified after maintenance or injection of the fresh calibration standard, re-calibrate the instrument.

11.3 The method blank must be extracted and analyzed with each set of samples at a frequency of at least 5% and must be free of the analytes of interest at the method detection limit. If the method blank contains target analytes at a detectable concentration, it may be necessary to analyze a solvent blank to demonstrate the source of the contamination is not carryover from standards or lingering field sample artifacts. Following verification that the analytical system is free from interferences, the method blank can be re-analyzed once. A passing method blank must be analyzed before any samples are analyzed; otherwise corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy or instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration. The surrogate recoveries in the method blank must meet the established

control criteria (see the LIMS). If not, the recovery demonstrates an analytical system that is in an out-of-control mode and the batch must be re-extracted/re-analysis unless directed otherwise by the client.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

11.4 Second Source Calibration Verification

Method 8270D/E: The value determined from the second source calibration verification (SSCV) should be within ±30% drift of the expected concentration. Alternative recovery limits may be appropriate based on analyte performance and project specific requirements. Quantitative analysis cannot proceed for analytes that fail the SSCV, except for screening purposes or estimated values only.

Method 8270C/SM6410B: The value determined from the second source calibration verification (SSCV) must be ≤50% drift for non-CCC compounds; ≤20% drift for CCC compounds and meet the minimum response factor criteria for SPCC compounds as in the initial calibration construction. Historical performance weighs heavily in the acceptability of those analytes that are known to perform poorly. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the SSCV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

STATE NOTE: If the samples are analyzed in conjunction with South Carolina DHEC, alternate recovery limits can only be used if they are more stringent than method criteria.

Method 625.1: Method 625.1 utilizes CCV limits for evaluation.

11.5 Surrogates: If the surrogate recoveries in the samples do not fall within the appropriate acceptance criteria presented in the LIMS, ensure that there were no errors in calculations, internal standard, or instrument performance. If the recovery of any one surrogate is critically low (, then the sample must be re-extracted unless otherwise directed by the client or a clear, documented matrix interference is exhibited. If two of three acid and two of three base/neutral surrogates are within acceptance criteria, then the sample may be reported. If surrogate recoveries present a high bias, and samples are BDL, then data is not impacted and may be reported. If re-extraction is required and there is no more sample available or it has exceeded holding times, the data must be flagged with a “J1" (surrogate high) or a “J2” (surrogate low). See SOP #030201, *Data Handling and Reporting*, for more information on qualifying out of control data.

STATE NOTE: If the sample is from North Carolina, two of the three acid and two of the three base/neutral surrogates must pass. If two of the three acid or base/neutral surrogates fail, the sample must be re-extracted. For all other samples, one of the three surrogates must pass from both the acid and base/neutral sides. If any surrogates have less than a 10% or greater than 200% recovery, and matrix interferences are not confirmed as the cause of the failure, the sample must be re-extracted.

STATE NOTE: If field samples are analyzed in conjunction with the Ohio VAP program, surrogate outliers in batch QC samples, including the method blank, LCS/LCSD, MS/MSD require re-extraction of the entire batch, if sufficient volume has been submitted by the client and an obvious matrix interferent is not present.

STATE NOTE: If the sample is analyzed in conjunction with the Ohio VAP, corrective action for failing QC (i.e. method blank, surrogate, MS/MSD, LCS/LCSD, ISTD, etc.) must be performed prior to flagging data, if sufficient sample volume was submitted by the client. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related.

11.6 Internal Standard: The internal standard area counts must be monitored for all ICVs. ISTDs must recover within –50% to +100% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take the form of checking the original calculations to ensure accuracy, re-analysis of the ICV to verify initial results, instrument maintenance (i.e. column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards must be monitored for each sample. ISTDs in samples must meet the –50% to +100% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than +100%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results. ISTD failures <50% of daily ICV may be reported if all corresponding analytes are BDL as the high quantitation bias created by the reduced internal standard recovery has not adversely impacted the reported analyte results.

11.7 LCS/LCSD and MS/MSD: The laboratory control sample, laboratory control sample duplicate, matrix spike and matrix spike duplicate recoveries must be evaluated against the acceptance criteria given in the LIMS. The LCS/LCSD and MS/MSD are spiked with the same list of compounds for which the instrument is calibrated. Due to the large

number of compounds analyzed using these methods, it is statistically likely that accuracy and precision failures will occur.

LCS or LCSD samples that do not pass the acceptable QC criteria must be re-analyzed. LCS/LCSD failures must meet the marginal exceedance criteria below. The normal compound list for 8270/625 typically contains 90 analytes; therefore only 5 analytes can be considered as marginally exceeding the acceptance criteria. If more than 5 failures occur or if the failures demonstrate a pattern that is causing the outliers, the entire sample batch with associated QC must be re-extracted and re-analyzed. Marginal exceedances must be random events.

Upper and lower marginal exceedance (ME) limits are established by +/- four times the standard deviation of historical accuracy data and the number of marginal exceedances allowed is based on the number of analytes spiked in the LCS.

Number of allowable marginal exceedances:

90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit.
51 – 70 analytes, 3 analytes allowed in the ME limit.
31 – 50 analytes, 2 analytes allowed in the ME limit.
11 – 30 analytes, 1 analyte allowed in the ME limit.
< 11 analytes, no analyte allowed in the ME limit.

If the MS/MSD fails to meet recovery limits listed in the LIMS, the data on the unspiked field sample for that compound must be flagged with a "J5" (high recovery) or a "J6" (low recovery). If the MS/MSD fail to pass precision limits (%RSD), the data on the unspiked field sample for that compound must be flagged with a "J3" qualifier.

STATE NOTE: For South Carolina or Ohio VAP samples, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

STATE NOTE: For all samples from South Carolina, the LCS/LCSD recovery must be evaluated within 70-130% and the MS/MSD recoveries must be within in-house derived recovery limits; however, if the limits given in Method 625 Table 6 are more stringent, then those limits must be used. The following are the current limits:

Parameter	LCS/LCSD	MS/MSD
1,2,4-TRICHLOROBENZENE	70 - 130%	44 – 104%
2,4,6-TRICHLOROPHENOL	70 - 130%	37 - 132%
2,4-DICHLOROPHENOL	70 - 130%	39 - 117%
2,4-DIMETHYLPHENOL	70 - 119%	32 - 119%
2,4-DINITROPHENOL	70 - 130%	10 - 141%
2,4-DINITROTOLUENE	70 - 130%	45.4 - 139%
2,6-DINITROTOLUENE	70 - 130%	50 - 134%
2-CHLORONAPHTHALENE	70 - 118%	60 - 118%
2-CHLOROPHENOL	70 - 130%	23 - 111%
2-NITROPHENOL	70 - 130%	29 - 135%
3,3-DICHLOROBENZIDINE	70 - 130%	10 - 143%
4,6-DINITRO-2-METHYLPHENOL	70 - 130%	10 - 143%
4-BROMOPHENYL-PHENYLETHER	70 - 127%	53 - 127%
4-CHLORO-3-METHYLPHENOL	70 - 130%	38.4 - 123%
4-CHLOROPHENYL-PHENYLETHER	70 - 130%	49.8 -127%

Parameter	LCS/LCSD	MS/MSD
4-NITROPHENOL	70 - 130%	10 - 52.8%
ACENAPHTHENE	70 - 130%	47 - 141%
ACENAPHTHYLENE	70 - 130%	40 - 132%
ANTHRACENE	70 - 130%	44.5 - 130%
BENZO(A)ANTHRACENE	70 - 130%	46.4 - 130%
BENZO(A)PYRENE	70 - 130%	34.6 - 129%
BENZO(B)FLUORANTHENE	70 - 130%	36.3 - 137%
BENZO(G,H,I)PERYLENE	70 - 130%	10 - 140%
BENZO(K)FLUORANTHENE	70 - 130%	30.3 - 136%
BENZYLBUTYL PHTHALATE	70 - 130%	44.8 - 152%
BIS(2-CHLORETHOXY)METHANE	70 - 130%	39.2 - 128%
BIS(2-CHLOROETHYL)ETHER	70 - 130%	14.8 - 131%
BIS(2-CHLOROISOPROPYL)ETHER	70 - 130%	36 - 117%
BIS(2-ETHYLHEXYL)PHTHALATE	70 - 130%	12.6 - 153%
CHRYSENE	70 - 130%	40 - 133%
DIBENZ(A,H)ANTHRACENE	70 - 130%	10 - 143%
DIETHYL PHTHALATE	70 - 114%	50.4 - 114%
DIMETHYL PHTHALATE	70 - 112%	9.1 - 112%
DI-N-BUTYL PHTHALATE	70 - 118%	53.3 - 118%
DI-N-OCTYL PHTHALATE	70 - 130%	13.3 - 146%
FLUORANTHENE	70 - 130%	42.9 - 137%
FLUORENE	70 - 121%	59 - 121%
HEXACHLORO-1,3-BUTADIENE	70 - 116%	28.9 - 116%
HEXACHLOROBENZENE	70 - 130%	47 - 121%
HEXACHLOROCYCLOPENTADIENE	70 - 130%	10 - 128%
HEXACHLOROETHANE	70 - 113%	40 - 109%
INDENO(1,2,3-CD)PYRENE	70 - 130%	10 - 141%
ISOPHORONE	70 - 130%	31.9 - 118%
NAPHTHALENE	70 - 130%	29 - 115%
NITROBENZENE	70 - 130%	35 - 118%
N-NITROSODI-N-PROPYLAMINE	70 - 130%	35.4 - 129%
PENTACHLOROPHENOL	70 - 130%	14 - 128%
PHENANTHRENE	70 - 130%	54 - 120%
PHENOL	70 - 112%	10 - 55.7%
PYRENE	70 - 115%	52 - 115%

Method 625.1: For LCS analyses, repeat the test only for those analytes that failed to meet the acceptance criteria (PS). If these analytes now pass, system performance is acceptable and analysis of blanks and samples may proceed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to analysis of the LCS prepared with a fresh sample concentrate, or to system repair, repeat the LCS test. If failure of the LCS indicates a systemic problem with samples in the batch, re-extract and re-analyze the samples in the batch.

The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results

associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

NOTE: To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests.

For MS/MSD analyses, compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in Attachment VII. A laboratory may develop and apply QC acceptance criteria more restrictive than the criteria in Attachment VII, if desired.

If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect. The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a retest is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

11.8 Calibration Range: For any compound found in a sample at a level above the highest standard, the extract must be diluted and re-analyzed to allow quantitation within the range of instrument calibration. Whenever an extract dilution is made, the appropriate amount of internal standard must be added to bring the ISTD concentrations back to the concentrations consistent with the calibration standards.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

11.9 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed, if necessary. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

11.10 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method.

11.10.1 The analyst should must review any sample that has quantifiable compounds and make sure that they have been confirmed.

11.10.2 All surrogate recoveries must be checked to ensure that they are within QC acceptance criteria or that corrective action has occurred.

11.10.3 Blanks must be free of all interfering peaks.

11.10.4 Quality control criteria must be checked for the LCS, LCSD, MS, and MSD.

11.10.5 Data must be checked to determine the need for appropriate flags. Comments are noted when results are flagged.

11.10.7 All manual integrations must be verified through checking the before/after shot of the sample, method blank, and/or QC (LCS/LCSD/MS/MSD).

11.10.8 See SOP #030201, *Data Handling and Reporting* and SOP #030227*, Data Review*.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051 *Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 The **Missouri Department of Natural Resources requires** that **DRO** be analyzed by GC/MS. Tuning and frequency requirements are the same as 8270C, omitting DDT, pentachlorophenol, and benzidine assessments. Extract samples the same as 8270PAH using the appropriate extraction method. Only base/neutral surrogates are needed. GC/MS mass range should be 35-550amu. Prepare a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 10,000 µg/mL each in methylene chloride. Calibration standards range from 200 to 10,000ug/mL for concentrated soil or 1L water extractions and calibration levels for EPA 3511 extracted water samples and non-concentrated Soil range from 5-200ppm from a 200ppm intermediate. Retention time windows are set using C_{10}, C_{21}, and C_{35}. For DRO, set RT 0.1 minutes after C_{10} to 0.1 minutes after C_{21}. For ORO, set RT 0.1 minutes after C_{21} to 0.1 minutes after C_{35}. Verify RT windows daily (24 hours) by running component standard. Quantitate using baseline-to baseline, not valley–to-valley. The total ion chromatogram must be used to quantitate. DRO is quantitated using external standard method. The response factor determined for DRO (C_{10}-C_{21}) **must** be used for C_{21}-C_{35}. Subtract area for any internal standard and surrogates. %RSD <20. Run a CCV every 12 hours near mid-point of calibration, %D <20. Run a method blank, LCS and MS/MSD every extraction batch. May re-process files to quantitate PAH analytes, if needed. Quantitation of DRO must be performed using the external standard process.

13.4 The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA 3510C/625method. The reduction in

volume extracted along with increased sensitivity at detection and/or analysis of the resulting extract using large volume injection (>5uL) on each GCMS allows for low detection limits typical of those obtained using a 1L extraction. Complete method validation is performed for each method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

13.5 **Low level NDMA and 1,4-Dioxane by SIM scan/isotope dilution.** Tuning and frequency requirements are the same as 8270C. Extract samples the same as 8270BNA using the appropriate extraction method. 250ng of N-nitrosodimethylamine-d6 or 1,4Dioxane-d_8 is added to each sample per every 0.5mL of final extract volume prior to extraction resulting in a true value of 500ppb in extract. Only base/neutral surrogates monitoring is necessary. GC/MS is set to scan for masses 42, 43, 46, 48, 54, 74, 80, 82, 115, 128, 150 and 152 for NDMA-d_6 or masses 57, 58, 62, 64, and 88 for 1,4-Dioxane-d_8 in SIM mode. Calibrate at least 5 points using 8270BNA mega mix or 1,4-Dioxane ICAL standard. 500ng of N-nitrosodimethylamine-d_6 is added per every 1mL of calibration standard to each level of the calibration resulting in a true value of 500ppb. Calibration standards range from 5ppb to 10,000ppb for 3510RV extracted water samples. Quantitate using Chemstation auto-integration software unless a significant discrepancy is noted in which case manually adjust integrations to best represent the calibration. Select ion monitoring should be used for acquisition and quantitation. NDMA and 1,4-Dioxane are quantitated using the isotope dilution method as described in 8000C. The %RSD determined for NDMA RFs **must** be <15% in order to use the average of response factors for quantitation, otherwise linear regression is to be used. Run a DFTPP tune and CCV every 12 hours near mid-point of calibration, %Diff must be <20% for the calibration to be deemed in control and sample analysis to proceed. Run a method blank, LCS and LCSD with every extraction batch. MS/MSDs will be processed with batches when requested by the client as matrix spiking and duplication does not yield reliable precision data when analyzed by the isotope dilution method. Quantitation of low level NDMA and 1,4-Dioxane should be performed using the isotope dilution process.

13.6 **Tetraethyllead by SIM analysis**. Tetraethyllead is analyzed by standard SIM operating procedures. Ions 237 and 295 are added to the routine SIM acquisition methods in SIM group 1. All calibration and analytical procedures are mirrored from analysis of PAH by SIM.

14.0 REFERENCES

14.1 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270C, Revision 3, December 1996.

14.2 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270D, Revision 4, February 2007.

14.3 *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW846 Method 8270E, Revision 6, February 2017.

14.4 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, December 1996.

14.5 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003.

14.6 *Determinative Chromatographic Separations*, SW846 Method 8000D, Revision 4, July 2014.

14.6 *Base/Neutrals and Acids*, 40 CFR Part 136, Appendix A, EPA Method 625, October 1991.

14.7 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-2000.

14.8 *Extractable Base/Neutrals and Acids*, Standard Methods for the Examination of Water and Wastewater, Method 6410B-1997 (20th Ed).

14.9 *Base/Neutrals and Acids by GC/MS*, EPA Method 625.1, Federal Register, Volume 82, Number 165, August 28, 2017.

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Semivolatile Organics by GCMS using Capillary Column (EPA Methods 8270C, D, E & 625.1) Including Provisions for Analysis in SIM Mode
ISSUER: Pace National – Mt. Juliet, Tennessee

Attachment I: Revision History

This Version:

Section	Description of Change
Updated sections: 1.0, 1.8, 1.8.1, 2.1, 4.7, 5.3, 7.6.26, 7.6.27, 8.2, 8.8, 8.9.1.1, 10.1, 10.1.1.2, 10.2, 12.1, 12.2, title, & revision history. Removed 4.2. Added 8.4.5, 8.4.5.1, 8.4.5.2.	Method validation update as well as minor revisions.

This document supersedes the following document(s):

Document Number	Title	Version
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	0
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	1
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	2
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	3
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	4
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	5
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	6
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	7
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	8

ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	9
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	10
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	11
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	12
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	13
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	14
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	15
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	16
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	17
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	18
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	19
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	20
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	21
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	22
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	23

ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	24
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	25
ESC Lab Sciences SOP #330345	ESC Lab Sciences SOP #330345	26
ENV-SOP-MTJL-0081	Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode	01
ENV-SOP-MTJL-0081	Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode	02
ENV-SOP-MTJL-0081	Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode	03
ENV-SOP-MTJL-0081	Semi-volatile Organics by GCMS using Capillary Column (EPA Methods 8270C, EPA 8270D, EPA Method 625, SM 6410B), Including Provisions for Analysis in SIM Mode	04

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Attachment II: 8270/625 Common Calibration List & Reporting Limits *(may be updated without notice)**

Analyte	Water mg/L	Soil mg/Kg
Acenaphthene	0.001	0.033
Acenaphthylene	0.001	0.033
Acetophenone	0.01	0.33
Anthracene	0.001	0.033
Atrazine	0.01	0.33
Benzaldehyde	0.01	0.33
Benzidine	0.05	0.33
Benzo(a)anthracene	0.001	0.033
Benzo(b)fluoranthene	0.001	0.033
Benzo(k)fluoranthene	0.001	0.033
Benzo(g,h,i)perylene	0.001	0.033
Benzo(a)pyrene	0.001	0.033
Bis(2-chlorethoxy)methane	0.01	0.33
Bis(2-chloroethyl)ether	0.01	0.33
Bis(2-chloroisopropyl)ether	0.01	0.33
4-Bromophenyl-phenylether	0.01	0.33
Caprolactam	0.01	0.33
2-Chloronaphthalene	0.01	0.33
4-Chlorophenyl-phenylether	0.01	0.33
Chrysene	0.001	0.033
Dibenz(a,h)anthracene	0.001	0.033
3,3-Dichlorobenzidine	0.01	0.33
2,4-Dinitrotoluene	0.01	0.33
2,6-Dinitrotoluene	0.01	0.33
Fluoranthene	0.001	0.033
Fluorene	0.001	0.033
Hexachlorobenzene	0.01	0.33
Hexachloro-1,3-butadiene	0.01	0.33
Hexachlorocyclopentadiene	0.01	0.33
Hexachloroethane	0.01	0.33
Indeno(1,2,3-cd)pyrene	0.001	0.033
Isophorone	0.01	0.33
Naphthalene	0.001	0.033
Nitrobenzene	0.01	0.33
n-Nitrosodimethylamine	0.01	0.33
n-Nitrosodiphenylamine	0.01	0.33
n-Nitrosodi-n-propylamine	0.01	0.33
Phenanthrene	0.001	0.033

Analyte	Water mg/L	Soil mg/Kg
Benzylbutyl phthalate	0.003	0.033
Bis(2-ethylhexyl)phthalate	0.003	0.033
Di-n-butyl phthalate	0.003	0.033
Diethyl phthalate	0.003	0.033
Dimethyl phthalate	0.003	0.033
Di-n-octyl phthalate	0.003	0.033
Pyrene	0.001	0.033
1,2,4-Trichlorobenzene	0.01	0.33
4-Chloro-3-methylphenol	0.01	0.33
2-Chlorophenol	0.01	0.33
2,4-Dichlorophenol	0.01	0.33
2,4-Dimethylphenol	0.01	0.33
4,6-Dinitro-2-methylphenol	0.01	0.33
2,4-Dinitrophenol	0.01	0.33
2-Methylphenol	0.01	0.33
4-Methylphenol	0.01	0.33
2-Nitrophenol	0.01	0.33
4-Nitrophenol	0.01	0.33
Pentachlorophenol	0.01	0.33
Phenol	0.01	0.33
2,4,6-Trichlorophenol	0.01	0.33
1-Methylnapthalene	0.001	0.033
2-Methylnapthalene	0.001	0.033
4-Chloroaniline	0.01	0.33
2-Nitroaniline	0.01	0.33
3-Nitroaniline	0.01	0.33
4-Nitroaniline	0.01	0.33
1,2,3,4-Tetrachlorobenzene	0.05	1.65
1,2,3,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2,4,5-Tetrachlorobenzene	0.05	1.65
1,2-diphenylhydrazine	0.01	0.33
1,3-Dinitrobenzene	0.05	1.65
1,4-Naphthoquinone	0.05	1.65
1-Chloronaphthalene	0.05	1.65
1-Naphthylamine	0.05	1.65
2,3,4,6-Tetrachlorophenol	0.05	1.65
2,3-Dichloroaniline	0.01	0.33

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Analyte	Water mg/L	Soil mg/Kg
2,6-Dichlorophenol	0.05	1.65
2-Acetylaminofluorene	0.05	1.65
2-Naphthylamine	0.05	1.65
2-Picoline	0.05	1.65
3,3'-Dimethylbenzidine	0.05	1.65
3-Methylcholanthrene	0.05	1.65
4-Aminobiphenyl	0.05	1.65
4-Nitroquinoline-1-oxide	0.05	1.65
5-Nitro-o-toluidine	0.05	1.65
7,12-Dimethylbenz(a)anthracene	0.05	1.65
7H-Dibenzo (c,g) carbazole	0.05	1.65
a,a-Dimethylphenethylamine	0.05	1.65
Acetophenone	0.01	0.33
Alpha-terpineol	0.01	0.33
Aniline	0.01	0.33
Aramite	0.05	1.65
Benzal Chloride	0.05	1.65
Benzo (j) fluoranthene	0.05	1.65
Benzotrichloride	0.05	1.65
Benzyl Chloride	0.05	1.65
Chlorobenzilate	0.05	1.65
Diallate (cis)	0.05	1.65
Diallate (trans)	0.05	1.65
Dibenz (a,e) pyrene	0.05	1.65
Dibenz (a,h) acridine	0.05	0.33
Dibenz (a,h) pyrene	0.05	1.65
Dibenz (a,i) pyrene	0.05	1.65
Dimethoate	0.05	1.65
Dinoseb	0.05	1.65
Diphenylamine	0.05	1.65
Disulfoton	0.05	1.65
Ethyl methanesulfonate	0.05	1.65
Famphur	0.05	1.65
Hexachlorophene	0.05	1.65
Hexachloropropene	0.05	1.65
Isodrin	0.05	1.65
Isosafrole (cis)	0.05	1.65
Isosafrole (trans)	0.05	1.65
Kepone	0.05	1.65

Analyte	Water mg/L	Soil mg/Kg
Methapyriline	0.05	1.65
Methyl methanesulfonate	0.05	1.65
Methyl parathion	0.05	1.65
N-Nitrosodiethylamine	0.05	1.65
n-nitrosodi-n-butylamine	0.01	0.33
N-Nitrosodi-n-butylamine	0.05	1.65
N-Nitrosomethylethylamine	0.05	1.65
N-Nitrosomorpholine	0.05	1.65
N-Nitrosopiperidine	0.05	1.65
N-Nitrosopyrrolidine	0.05	1.65
o,o,o-Triethylphoshorothioate	0.05	1.65
o-cresol	0.01	0.33
o-Toluidine	0.05	1.65
Parathion	0.05	1.65
p-cresol	0.01	0.33
p-Dimethylaminoazobenzene	0.05	1.65
Pentachlorobenzene	0.05	1.65
Pentachloroethane	0.05	1.65
Pentachloronitrobenzene	0.05	1.65
Phenacetin	0.05	1.65
Phorate	0.05	1.65
p-Phenyleneamine	0.05	1.65
Pronamide	0.05	1.65
Safrole	0.05	1.65
Sulfotepp	0.05	1.65
sym-Trinitrobenzene	0.05	1.65
Thionazin	0.05	1.65
2-nitrodiphenylamine	0.01	0.33
n-decane	0.01	0.33
n-octadecane	0.01	0.33
Pentachlorphenol (SIM)	0.001	-
Sulfolane	0.0002	0.33
Mirex	0.02	NA
Dicofol	0.02	NA
Quinoline	0.05	0.33
Indene	NA	0.33
Benzenethiol	0.02	3.3

*****Alternate reporting levels may be possible using different technologies (i.e. SIM, LVI, etc.).

Please contact the laboratory for additional information.

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Attachment III – Appropriate Extraction Methods by Analyte *(printed from SW-846 Method 8270C)*

ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Acenaphthene	X	X	X	X	X	83-32-9
Acenaphthylene	X	X	X	X	X	208-96-8
Acetophenone	X	ND	ND	ND	X	98-86-2
2-Acetylaminofluorene	X	ND	ND	ND	X	53-96-3
1-Acetyl-2-thiourea	LR	ND	ND	ND	LR	591-08-2
Aldrin	X	X	X	X	X	309-00-2
2-Aminoanthraquinone	X	ND	ND	ND	X	117-79-3
Aminoazobenzene	X	ND	ND	ND	X	60-09-3
4-Aminobiphenyl	X	ND	ND	ND	X	92-67-1
3-Amino-9-ethylcarbazole	X	X	ND	ND	ND	132-32-1
Anilazine	X	ND	ND	ND	X	101-05-3
Aniline	X	X	ND	X	X	62-53-3
Ortho-anisidine	X	ND	ND	ND	X	90-04-0
Anthracene	X	X	X	X	X	120-12-7
Aramite HS	(43)	ND	ND	ND	X	140-57-8
Aroclor 1016	X	X	X	X	X	12674-11-2
Aroclor 1221	X	X	X	X	X	11104-28-2
Aroclor 1232	X	X	X	X	X	11141-16-5
Aroclor 1242	X	X	X	X	X	53469-21-9
Aroclor 1248	X	X	X	X	X	12672-29-6
Aroclor 1254	X	X	X	X	X	11097-69-1
Aroclor 1260	X	X	X	X	X	11096-82-5
Azinphos-methyl HS	(62)	ND	ND	ND	X	86-50-0
Barban	LR	ND	ND	ND	LR	101-27-9
Benzidine	CP	CP	CP	CP	CP	92-87-5
Benzoic Acid	X	X	ND	X	X	65-85-0
Benz(a)anthracene	X	X	X	X	X	56-55-3
Benzo(b)fluoranthene	X	X	X	X	X	205-99-2
Benzo(k)fluoranthene	X	X	X	X	X	207-08-9
Benzo(g,h,i)perylene	X	X	X	X	X	191-24-2
Benzo(a)pyrene	X	X	X	X	X	50-32-8
Para-benzoquinone	OE	ND	ND	ND	X	106-51-4
Benzyl Alcohol	X	X	ND	X	X	100-51-6
Alpha-BHC	X	X	X	X	X	319-84-6
Beta-BHC	X	X	X	X	X	319-85-7
Delta-BHC	X	X	X	X	X	319-86-8
Gamma-BHC	X	X	X	X		58-89-9
Lindane	X	X	X	X	X	58-89-9
Bis(2-chloroethoxy)methane	X	X	X	X	X	111-91-1
Bis(2-chloroethyl) Ether	X	X	X	X	X	111-44-4
Bis(2-chloroisopropyl) Ether	X	X	X	X	X	108-60-1
Bis(2-ethylhexyl) Phthalate	X	X	X	X	X	117-81-7
4-Bromophenyl Phenyl Ether	X	X	X	X	X	101-55-3
Bromoxynil	X	ND	ND	ND	X	1689-84-5
Butyl Benzyl Phthalate	X	X	X	X	X	85-68-7

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Captafol HS	(55)	ND	ND	ND	X	6/1/2425
Captan HS	(40)	ND	ND	ND	X	133-06-2
Carbaryl	X	ND	ND	ND	X	63-25-2
Carbofuran	X	ND	ND	ND	X	1563-66-2
Carbophenothion	X	ND	ND	ND	X	786-19-6
Chlordane	X	X	X	X	X	57-74-9
Chlorfenvinphos	X	ND	ND	ND	X	470-90-6
4-Chloroaniline	X	ND	ND	ND	X	106-47-8
Chlorobenzilate	X	ND	ND	ND	X	510-15-6
5-Chloro-2-methylaniline	X	ND	ND	ND	X	95-79-4
4-Chloro-3-methylphenol	X	X	X	X	X	59-50-7
hydrochloride	X	ND	ND	ND	X	6959-48-4
1-Chloronaphthalene	X	X	X	X	X	90-13-1
2-Chloronaphthalene	X	X	X	X	X	91-58-7
2-Chlorophenol	X	X	X	X	X	95-57-8
4-Chloro-1,2-phenylenendiamine	X	X	ND	ND	ND	95-83-0
4-Chloro-1,3-phenylenendiamine	X	X	ND	ND	ND	5131-60-2
4-Chlorophenyl Phenyl Ether	X	X	X	X	X	7005-72-3
Chrysene	X	X	X	X	X	218-01-9
Coumaphos	X	ND	ND	ND	X	56-72-4
Para-cresidine	X	ND	ND	ND	X	120-71-8
Crotoxyphos	X	ND	ND	ND	X	7700-17-6
2-Cyclohexyl-4,6-dinitrophenol	X	ND	ND	ND	LR	131-89-5
4,''-DDD	X	X	X	X	X	72-54-8
4,''-DDE	X	X	X	X	X	72-55-9
4,''-DDT	X	X	X	X	X	50-29-3
Demeton-O HS	(68)	ND	ND	ND	X	298-03-3
Demeton-S	X	ND	ND	ND	X	126-75-0
Diallate (cis or trans)	X	ND	ND	ND	X	2303-16-4
2,4-Diaminotoluene DC,	OE(42) ND	ND	ND	ND	X	95-80-7
Dibenz(a,j)acridine	X	ND	ND	ND	X	224-42-0
Dibenz(a,h)anthracene	X	X	X	X	X	53-70-3
Dibenzofuran	X	X	ND	X	X	132-64-9
Dibenzo(a,e)pyrene	ND	ND	ND	ND	X	192-65-4
1,2-Dibromo-3-chloropropane	X	X	ND	ND	ND	96-12-8
Di-n-butyl Phthalate	X	X	X	X	X	84-74-2
Dichlone	OE	ND	ND	ND	X	117-80-6
1,2-Dichlorobenzene	X	X	X	X	X	95-50-1
1,3-Dichlorobenzene	X	X	X	X	X	541-73-1
1,4-Dichlorobenzene	X	X	X	X	X	106-46-7
3,3''-Dichlorobenzidine	X	X	X	X	X	91-94-1
2,4-Dichlorophenol	X	X	X	X	X	120-83-2
2,6-Dichlorophenol	X	ND	ND	ND	X	87-65-0
Dichlorovos	X	ND	ND	ND	X	62-73-7
Dicrotophos	X	ND	ND	ND	X	141-66-2
Dieldrin	X	X	X	X	X	60-57-1

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Diethyl Phthalate	X	X	X	X	X	84-66-2
Diethylstilbestrol	AW,OS(67)	ND	ND	ND	X	56-53-1
Diethyl Sulfate	LR	ND	ND	ND	LR	64-67-5
Dihydrosaffrole	ND	ND	ND	ND	ND	56312-13-1
Dimethoate	HE,HS	ND	ND	ND	X	60-51-5
3,''-Dimethoxybenzidine	X	ND	ND	ND	LR	119-90-4
Dimethylaminoazobenzene	X	ND	ND	ND	X	60-11-7
7,12-Dimethylbenz(a)-anthracene	CP(45)	ND	ND	ND	CP	57-97-6
3,''-Dimethylbenzidine	X	ND	ND	ND	X	119-93-7
α,α-Dimethylphenethylamine	ND	ND	ND	ND	X	122-09-8
2,4-Dimethylphenol	X	X	X	X	X	105-67-9
Dimethyl Phthalate	X	X	X	X	X	131-11-3
1,2-Dinitrobenzene	X	ND	ND	ND	X	528-29-0
1,3-Dinitrobenzene	X	ND	ND	ND	X	99-65-0
1,4-Dinitrobenzene	HE(14)	ND	ND	ND	X	100-25-4
4,6-Dinitro-2-methylphenol	X	X	X	X	X	534-52-1
2,4-Dinitrophenol	X	X	X	X	X	51-28-5
2,4-Dinitrotoluene	X	X	X	X	X	121-14-2
2,6-Dinitrotoluene	X	X	X	X	X	606-20-2
Dinocap	CP,HS(28)	ND	ND	ND	CP	39300-45-3
Dinoseb	X	ND	ND	ND	X	88-85-7
Dioxathion	ND	ND	ND	ND	ND	78-34-2
Diphenylamine	X	X	X	X	X	122-39-4
5,5-Diphenylhydantoin	X	ND	ND	ND	X	57-41-0
1,2-Diphenylhydrazine	X	X	X	X	X	122-66-7
Di-n-octyl Phthalate	X	X	X	X	X	117-84-0
Disulfoton	X	ND	ND	ND	X	298-04-4
Endosulfan I	X	X	X	X	X	959-98-8
Endosulfan II	X	X	X	X	X	33212-65-9
Endosulfan Sulfate	X	X	X	X	X	1031-07-8
Endrin	X	X	X	X	X	72-20-8
Endrin Aldehyde	X	X	X	X	X	7421-93-4
Endrin Ketone	X	X	ND	X	X	53494-70-5
EPN	X	ND	ND	ND	X	2104-64-5
Ethion	X	ND	ND	ND	X	563-12-2
Ethyl Carbamate	DC(28)	ND	ND	ND	X	51-79-6
Ethyl Methanesulfonate	X	ND	ND	ND	X	62-50-0
Famphur	X	ND	ND	ND	X	52-85-7
Fensulfothion	X	ND	ND	ND	X	115-90-2
Fenthion	X	ND	ND	ND	X	55-38-9
Fluchloralin	X	ND	ND	ND	X	33245-39-5
Fluoranthene	X	X	X	X	X	206-44-0
Fluorene	X	X	X	X	X	86-73-7
2-Fluorobiphenyl (Surr)	X	X	X	X	X	321-60-8
2-Fluorophenol (Surr)	X	X	X	X	X	367-12-4
Heptachlor	X	X	X	X	X	76-44-8

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Heptachlor Epoxide	X	X	X	X	X	1024-57-3
Hexachlorobenzene	X	X	X	X	X	118-74-1
Hexachlorobutadiene	X	X	X	X	X	87-68-3
Hexachlorocyclopentadiene	X	X	X	X	X	77-47-4
Hexachloroethane	X	X	X	X	X	67-72-1
Hexachlorophene	AW,CP(62)	ND	ND	ND	CP	70-30-4
Hexachloropropene	X	ND	ND	ND	X	1888-71-7
Hexamethylphosphoramide	X	ND	ND	ND	X	680-31-9
Hydroquinone	ND	ND	ND	ND	X	123-31-9
Indeno(1,2,3-cd)pyrene	X	X	X	X	X	193-39-5
Isodrin	X	ND	ND	ND	X	465-73-6
Isophorone	X	X	X	X	X	78-59-1
Isosafrole	DC(46) ND	ND	ND	ND	X	120-58-1
Kepone	X	ND	ND	ND	X	143-50-0
Leptophos	X	ND	ND	ND	X	21609-90-5
Malathion	HS(5)	ND	ND	ND	X	121-75-5
Maleic Anhydride	HE	ND	ND	ND	X	108-31-6
Mestranol	X	ND	ND	ND	X	72-33-3
Methapyrilene	X	ND	ND	ND	X	91-80-5
Methoxychlor	X	ND	ND	ND	X	72-43-5
3-Methylcholanthrene	X	ND	ND	ND	X	56-49-5
4,''-Methylenebis (2-chloroaniline)	OE,OS(0)	ND	ND	ND	LR	101-14-4
4,''-Methylenebis-(N-n-dimethylaniline)	X	X	ND	ND	ND	101-61-1
Methyl methanesulfonate	X	ND	ND	ND	X	66-27-3
2-Methylnaphthalene	X	X	ND	X	X	91-57-6
Methyl Parathion	X	ND	ND	ND	X	298-00-0
2-Methylphenol	X	ND	ND	ND	X	95-48-7
3-Methylphenol	X	ND	ND	ND	X	108-39-4
4-Methylphenol	X	ND	ND	ND	X	106-44-5
2-Methylpyridine	X	X	ND	ND	ND	109-06-8
Mevinphos	X	ND	ND	ND	X	7786-34-7
Mexacarbate	HE,HS(68)	ND	ND	ND	X	315-18-4
Mirex	X	ND	ND	ND	X	2385-85-5
Monocrotophos	HE	ND	ND	ND	X	6923-22-4
Naled	X	ND	ND	ND	X	300-76-5
Naphthalene	X	X	X	X	X	91-20-3
1,4-Naphthoquinone	X	ND	ND	ND	X	130-15-4
1-Naphthylamine	OS(44)	ND	ND	ND	X	134-32-7
2-Naphthylamine	X	ND	ND	ND	X	91-59-8
Nicotine	DE(67)	ND	ND	ND	X	54-11-5
5-Nitroacenaphthene	X	ND	ND	ND	X	602-87-9
2-Nitroaniline	X	X	ND	X	X	88-74-4
3-Nitroaniline	X	X	ND	X	X	99-09-2
4-Nitroaniline	X	X	ND	X	X	100-01-6
5-Nitro-o-anisidine	X	ND	ND	ND	X	99-59-2

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Nitrobenzene	X	X	X	X	X	98-95-3
4-Nitrobiphenyl	X	ND	ND	ND	X	92-93-3
Nitrofen	X	ND	ND	ND	X	1836-75-5
2-Nitrophenol	X	X	X	X	X	88-75-5
4-Nitrophenol	X	X	X	X	X	100-02-7
5-Nitro-o-toluidine	X	ND	ND	ND	X	99-55-8
Nitroquinoline-1-oxide	X	ND	ND	ND	X	56-57-5
N-nitrosodi-n-butylamine	X	ND	ND	ND	X	924-16-3
N-nitrosodiethylamine	X	ND	ND	ND	X	55-18-5
N-nitrosodimethylamine	X	X	X	X	X	62-75-9
N-nitrosomethylethylamine	X	ND	ND	ND	X	10595-95-6
N-nitrosodiphenylamine	X	X	X	X	X	86-30-6
N-nitrosodi-n-propylamine	X	X	X	X	X	621-64-7
N-nitrosomorpholine	ND	ND	ND	ND	X	59-89-2
N-nitrosopiperidine	X	ND	ND	ND	X	100-75-4
N-nitrosopyrrolidine	X	ND	ND	ND	X	930-55-2
Octamethyl Pyrophosphoramide	LR	ND	ND	ND	LR	152-16-9
Parathion	X	ND	ND	ND	X	56-38-2
Pentachlorobenzene	X	ND	ND	ND	X	608-93-5
Pentachloronitrobenzene	X	ND	ND	ND	X	82-68-8
Pentachlorophenol	X	X	X	X	X	87-86-5
Phenacetin	X	ND	ND	ND	X	62-44-2
Phenanthrene	X	X	X	X	X	85-01-8
Phenobarbital	X	ND	ND	ND	X	50-06-6
Phenol	DC(28)	X	X	X	X	108-95-2
1,4-Phenylenediamine	X	ND	ND	ND	X	106-50-3
Phorate	X	ND	ND	ND	X	298-02-2
Phosalone	HS(65)	ND	ND	ND	X	2310-17-0
Phosmet	HS(15)	ND	ND	ND	X	732-11-6
Phosphamidon	HE(63)	ND	ND	ND	X	13171-21-6
Phthalic Anhydride	CP,HE(1)	ND	ND	ND	CP	85-44-9
2-Picoline	X	X	ND	ND	ND	109-06-8
Piperonyl Sulfoxide	X	ND	ND	ND	X	120-62-7
Pronamide	X	ND	ND	ND	X	23950-58-5
Pyrene	X	X	X	X	X	129-00-0
Pyridine	ND	ND	ND	ND	ND	110-86-1
Resorcinol	DC, OE(10)	ND	ND	ND	X	94-59-7
Safrole	X	ND	ND	ND	X	60-41-3
Sulfallate	X	ND	ND	ND	X	95-06-7
Terbufos	X	ND	ND	ND	X	13071-79-9
Terphenyl d(l4)(surr)	X	X	ND	X	X	1718-51-0
1,2,4,5-Tetrachlorobenzene	X	ND	ND	ND	X	95-94-3
2,3,4,6-Tetrachlorophenol	X	ND	ND	ND	X	58-90-2
Tetrachlorvinphos	X	ND	ND	ND	X	961-11-5
Tetraethyl Dithiopyrophosphate	X	X	ND	ND	ND	3689-24-5
Tetraethyl Pyrophosphate	X	ND	ND	ND	X	107-49-3

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ANALYTE:	3510*	3520	3540/3541	3550*	3580*	CAS #(a)
Thionazine	X	ND	ND	ND	X	297-97-2
Thiophenol	X	ND	ND	ND	X	108-98-5
Benzenethiol	X	ND	ND	ND	X	108-98-5
Toluene Diisocyanate	HE(6)	ND	ND	ND	X	584-84-9
Ortho-toluidine	X	ND	ND	ND	X	95-53-4
Toxaphene	X	X	X	X	X	8001-35-2
1,2,4-Trichlorobenzene	X	X	X	X	X	120-82-1
2,4,5-Trichlorophenol	X	X	ND	X	X	95-95-4
2,4,6-Trichlorophenol	X	X	X	X	X	88-06-2
Trifluralin	X	ND	ND	ND	X	1582-09-8
2,4,5-Trimethylaniline	X	ND	ND	ND	X	137-17-7
Trimethyl Phosphate	HE(60)	ND	ND	ND	X	512-56-1
1,3,5-Trinitrobenzene	X	ND	ND	ND	X	99-35-4
Tris(2,3-dibromopropyl) phosphate	X	ND	ND	ND	LR	126-72-7
O,O,O-Triethyl Phosphorothioate	X	ND	ND	ND	X	126-68-1

KEY TO ANALYTE LIST Pace National extraction technique Chemical Abstract Service Registry Number
(b) See Sec. 1.2 for other acceptable preparation methods.
(IS) This compound may be used as an internal standard.
(surr) This compound may be used as a surrogate.
(AW) Adsorption to walls of glassware during extraction and storage.
(CP) Non-reproducible chromatographic performance.
(DC) Unfavorable distribution coefficient (number in parenthesis is percent recovery).
(HE) Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
(HS) Hydrolysis during storage (number in parenthesis is percent stability).
(LR) Low response.
(ND) Not determined.
(OE) Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
(OS) Oxidation during storage (number in parenthesis is percent stability).
(X) Greater than 70 percent recovery by this technique.

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Attachment IV: Characteristic Masses (m/z) for Extractable Organic Compounds

(Reprinted from SW-846 Method 8270C /Dec. 1996)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52,78,51
N-Nitrosodimethylamine	42	74,44
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Benzaldehyde	105	106,77,51
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d4 (ISTD)	152	150,115
1,4-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77,121
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
Ethyl methanesulfonate	79	109,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d8 (ISTD)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56,42
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90
Hexachloropropene	213	211,215,117,106,141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
4-Methylphenol	107	108,77,79,90
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
o-Toluidine	106	107,77,51,79

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
3-Methylphenol	107	108,77,79,90
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1-Chloronaphthalene	162	127,164
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89
3-Nitroaniline	138	108,92
Acenaphthene-d10 (ISTD)	164	162,160
Acenaphthene	154	153,152
2,4-Dinitrophenol	184	63,154
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
4-Nitrophenol	139	109,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,50,130
Diethyl phthalate	149	177,150
Fluorene	166	165,167
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
Atrazine	200	215,58
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
1-Naphthylamine	143	115,89,63
4-Bromophenyl phenyl ether	248	250,141
2,4,5-Trichlorophenol	196	198,97,132,99
Hexachlorobenzene	284	142,249
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazine	107	96,97,143,79,68
4-Nitroaniline	138	65,108,92,80,39
Phenanthrene-d10 (ISTD)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166,168,139
1,3-Dinitrobenzene	168	76,50,75,92,122
Diallate (cis or trans)	86	234,43,70
Pentachlorobenzene	250	252,108,248,215,254
Pentachloronitrobenzene	237	142,214,249,295,265

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ISSUER: Pace National – Mt. Juliet, Tennessee

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
4-Nitroquinoline-1-oxide	174	101,128,75,116
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Demeton-O	88	89,60,61,115,171
Fluoranthene	202	101,203
1,3,5-Trinitrobenzene	75	74,213,120,91,63
Benzidine	184	92,185
Pyrene	202	200,203
Phorate	75	121,97,93,260
Demeton-S	88	60,81,89,114,115
Phenacetin	108	180,179,109,137,80
Dimethoate	87	93,125,143,229
4-Aminobiphenyl	169	168,170,115
Dimethylphenylamine	58	91,65,134,42
Pronamide	173	175,145,109,147
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Butyl benzyl phthalate	149	91,206
Methyl parathion	109	125,263,79,93
Dimethylaminoazobenzene	225	120,77,105,148,42
Benz(a)anthracene	228	229,226
Chrysene-d12 (ISTD)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Kepone	272	274,237,178,143,270
Parathion	109	97,291,139,155
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
Di-n-octyl phthalate	149	167,43
Aramite	185	191,319,334,197,321
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Famphur	218	125,93,109,217
Benzo(a)pyrene	252	253,125
Perylene-d12 (ISTD)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
2-Acetylaminofluorene	181	180,223,152
3-Methylcholanthrene	268	252,253,126,134,113
Dibenz(a,j)acridine	279	280,277,250
Indeno(1,2,3-cd)pyrene	276	138,227
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
Hexachlorophene	196	198,209,211,406,408

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Diphenylhydrazine/Azobenzene	77	105,182
Mirex	272	274, 237,270
Kelthane (Dicofol)	251	139.111.253
Indene	115	116,117
Quinoline	129	128,130,102
Benzenethiol	110	109,66
Diphenyl Disulfide	218	109,65
Surrogates		
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Nitrobenzene-d5 (surr)	82	128,54
Phenol-d6 (surr)	99	42,71
Terphenyl-d14 (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141
2-Methylnaphthalene-d10 (surr)	152	150, 122, 151
Fluoranthene-d10 (surr)	212	208, 313, 210
PAH by SIM		
Naphthalene	128	129
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
2-Chloronaphthalene	162	127
Acenaphthylene	152	153, 151
Acenaphthene	153	154, 152, 151
Dibenzofuran	168	139
Fluorene	166	165
Phenanthrene	178	179
Anthracene	178	179, 176
Fluoranthene	202	203, 200
Pyrene	202	203, 200
Benzo(a)anthracene	228	226
Chrysene	228	226, 229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	277, 138
Dibenz(a,h)anthracene	278	279, 139, 138
Benzo(g,h,i)perylene	276	138

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Attachment V - QC Acceptance Criteria for Method 625

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39	7.2-152.2	D-166
Anthracene	100	32	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39	31.7-148.0	17-163
Benzo(g,h,i)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
beta-BHC	100	31.5	41.5-130.6	24-149
delta-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23	64.9-114.4	53-127
2-Chloronaphthalene	100	13	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31	D-134.5	D-145
4,4'-DDE	100	32	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octyl phthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p(s) (%)
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Aroclor 1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

(s) = Standard deviation of four recovery measurements, in µg/L

(x) = Average recovery for four recovery measurements, in µg/L

(p, p(s)) = Measured percent recovery

(D) = Detected; result must be greater than zero

(a) = Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop method performance data. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

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Attachment VI - BNA Poor Performing Compounds

The following compounds are considered to be poor performing compounds.
Pyridine
Aniline
Benzoic Acid
n-Nitrosodimethylamine
Hexachlorocyclopentadiene
4-Chloroaniline
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
2,4-Dinitro-2-methylphenol
Pentachlorophenol
Carbazole
Benzidine
Atrazine
Acetophenone
Caprolactam
Benzaldehyde
1,2,4,5-Tetrachlorobenzene
Hexachlorophene

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Attachment VII – Method 625.1 Criteria

Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Acenaphthene*	70-130	29	60-132	47-145	48
Acenaphthylene*	60-130	45	54-126	33-145	74
Anthracene*	58-130	40	43-120	27-133	66
Benzidine*					
Benzo(a)anthracene*	42-133	32	42-133	33-143	53
Benzo(a)pyrene*	32-148	43	32-148	17-163	72
Benzo(b)fluoranthene*	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene*	25-146	38	25-146	11-162	63
Benzo(ghi)perylene*	13-195	61	D-195	D-219	97
Benzyl butyl phthalate*	43-140	36	D-140	D-152	60
bis(2-Chloroethoxy)methane	52-164	32	49-165	33-184	54
bis(2-Ethylhexyl)phthalate*	43-137	50	29-137	8-158	82
bis(2-Chloroisopropyl) ether (2,2'-Oxybis[1-chloropropane])*	63-139	46	63-139	36-166	76
4-Bromophenyl phenyl ether*	70-130	26	65-120	53-127	43
2-Chloronaphthalene*	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl ether*	57-145	36	38-145	25-158	61
Chrysene*	44-140	53	44-140	17-168	87
Dibenz(a,h)anthracene*	13-200	75	D-200	D-227	126
Di-n-butylphthalate*	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine*	18-213	65	8-213	D-262	108
Diethyl phthalate*	47-130	60	D-120	D-120	100
Dimethyl phthalate*	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene*	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene*	68-137	29	68-137	50-158	48
Di-n-octylphthalate*	21-132	42	19-132	4-146	69
Fluoranthene*	47-130	40	43-121	26-137	66
Fluorene*	70-130	23	70-120	59-121	38
Hexachlorobenzene*	38-142	33	8-142	D-152	55
Hexachlorobutadiene*	68-130	38	38-120	24-120	62
Hexachloroethane*	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene*	13-151	60	D-151	D-171	99
Isophorone*	52-180	56	47-180	21-196	93
Naphthalene*	70-130	39	36-120	21-133	65
Nitrobenzene*	54-158	37	54-158	35-180	62
N-Nitrosodi-n-propylamine*	59-170	52	14-198	D-230	
Phenanthrene*	67-130	24	65-120	54-120	39
Pyrene*	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene*	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
2-Chlorophenol	55-130	37	36-120	23-134	61
2,4-Dichlorophenol	64-130	30	53-122	39-155	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2-Methyl-4,6-dinitrophenol	56-130	122	53-130	D-181	203
2-Nitrophenol	61-163	33	45-167	29-182	55

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4-Nitrophenol	35-130	79	13-129	D-132	131
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenol	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58
4-Chloro-3-methylphenol					
2-Chlorophenol					
2,4-Dichlorophenol					
2,4-Dimethylphenol					
2,4-Dinitrophenol					
2-Methyl-4,6-dinitrophenol					
2-Nitrophenol					
4-Nitrophenol					
Acetophenone					
2-Acetylaminofluorene					
1-Acetyl-2-thiourea					
Alachlor					
Aldrin	7-152	39	7-152	D-166	81
Ametryn					
2-Aminoanthraquinone					
Aminoazobenzene					
4-Aminobiphenyl					
3-Amino-9-ethylcarbazole					
Anilazine					
Aniline					
o-Anisidine					
Aramite					
Atraton					
Atrazine					
Azinphos-methyl					
Barban					
Benzanthrone					
Benzenethiol					
Benzoic acid					
2,3-Benzofluorene					
p-Benzoquinone					
Benzyl alcohol					
alpha-BHC					
beta-BHC	42-131	37	42-131	24-149	61
gamma-BHC (Lindane)					
delta-BHC	D-130	77	D-120	D-120	129
Biphenyl					
Bromacil					
2-Bromochlorobenzene					
3-Bromochlorobenzene					
Bromoxynil					
Butachlor					
Butylate					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
n-C10 (n-decane)					
n-C12 (n-undecane)					
n-C14 (n-tetradecane)					
n-C16 (n-hexadecane)					
n-C18 (n-octadecane)					
n-C20 (n-eicosane)					
n-C22 (n-docosane)					
n-C24 (n-tetracosane)					
n-C26 (n-hexacosane)					
n-C28 (n-octacosane)					
n-C30 (n-triacontane)					
Captafol					
Captan					
Carbaryl					
Carbazole					
Carbofuran					
Carboxin					
Carbophenothion					
Chlordane 3,5					
bis(2-Chloroethyl) ether	52-130	65	43-126	12-158	108
Chloroneb					
4-Chloroaniline					
Chlorobenzilate					
Chlorfenvinphos					
4-Chloro-2-methylaniline					
3-(Chloromethyl)pyridine hydrochloride					
4-Chloro-2-nitroaniline					
Chlorpropham					
Chlorothalonil					
1-Chloronaphthalene					
3-Chloronitrobenzene					
4-Chloro-1,2-phenylenediamine					
4-Chloro-1,3-phenylenediamine					
2-Chlorobiphenyl					
Chlorpyrifos					
Coumaphos					
m + p-Cresol					
o-Cresol					
p-Cresidine					
Crotoxyphos					
2-Cyclohexyl-4,6-dinitro-phenol					
Cyanazine					
Cycloate					
p-Cymene					
Dacthal (DCPA)					
4,4'-DDD	D-135	56	D-135	D-145	93

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
4,4'-DDE	19-130	46	19-120	4-136	77
4,4'-DDT	D-171	81	D-171	D-203	135
Demeton-O					
Demeton-S					
Diallate (cis or trans)					
2,4-Diaminotoluene					
Diazinon					
Dibenz(a,j)acridine					
Dibenzofuran					
Dibenzo(a,e)pyrene					
Dibenzothiophene					
1,2-Dibromo-3-chloropropane					
3,5-Dibromo-4-hydroxybenzonitrile					
2,6-Di-tert-butyl-p-benzoquinone					
Dichlone					
2,3-Dichloroaniline					
2,3-Dichlorobiphenyl					
2,6-Dichloro-4-nitroaniline					
2,3-Dichloronitrobenzene					
1,3-Dichloro-2-propanol					
2,6-Dichlorophenol					
Dichlorvos					
Dicrotophos					
Dieldrin 3	70-130	38	44-119	29-136	62
1,2:3,4-Diepoxybutane					
Di(2-ethylhexyl) adipate					
Diethylstilbestrol					
Diethyl sulfate					
Dilantin (5,5-Diphenylhydantoin)					
Dimethoate					
3,3′-Dimethoxybenzidine					
Dimethylaminoazobenzene					
7,12-Dimethylbenz(a)anthracene					
3,3′-Dimethylbenzidine					
N,N-Dimethylformamide					
3,6-Dimethylphenathrene					
alpha, alpha-Dimethylphenethylamine					
Dimethyl sulfone					
1,2-Dinitrobenzene					
1,3-Dinitrobenzene					
1,4-Dinitrobenzene					
Dinocap					
Dinoseb					
Diphenylamine					
Diphenyl ether					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
1,2-Diphenylhydrazine					
Diphenamid					
Diphenyldisulfide					
Disulfoton					
Disulfoton sulfoxide					
Disulfoton sulfone					
Endosulfan I					
Endosulfan II					
Endosulfan sulfate	D-130	42	D-120	D-120	70
Endrin					
Endrin aldehyde	D-189	45	D-189	D-209	75
Endrin ketone					
EPN					
EPTC					
Ethion					
Ethoprop					
Ethyl carbamate					
Ethyl methanesulfonate					
Ethylenethiourea					
Etridiazole					
Ethynylestradiol-3-methyl ether					
Famphur					
Fenamiphos					
Fenarimol					
Fensulfothion					
Fenthion					
Fluchloralin					
Fluridone					
Heptachlor	D-172	44	D-172	D-192	74
Heptachlor epoxide	70-130	61	71-120	26-155	101
2,2′,3,3′,4,4′,6-Heptachlorobiphenyl					
2,2′,4,4′,5′,6-Hexachlorobiphenyl					
Hexachlorocyclopentadiene					
Hexachlorophene					
Hexachloropropene					
Hexamethylphosphoramide					
Hexanoic acid					
Hexazinone					
Hydroquinone					
Isodrin					
2-Isopropylnaphthalene					
Isosafrole					
Kepone					
Leptophos					
Longifolene					
Malachite green					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Malathion					
Maleic anhydride					
Merphos					
Mestranol					
Methapyrilene					
Methoxychlor					
2-Methylbenzothioazole					
3-Methylcholanthrene					
4,4′-Methylenebis(2-chloroaniline)					
4,4′-Methylenebis(N,N-dimethylaniline)					
4,5-Methylenephenanthrene					
1-Methylfluorene					
Methyl methanesulfonate					
2-Methylnaphthalene					
Methylparaoxon					
Methyl parathion					
1-Methylphenanthrene					
2-(Methylthio)benzothiazole					
Metolachlor					
Metribuzin					
Mevinphos					
Mexacarbate					
MGK 264					
Mirex					
Molinate					
Monocrotophos					
Naled					
Napropamide					
1,4-Naphthoquinone					
1-Naphthylamine					
2-Naphthylamine					
1,5-Naphthalenediamine					
Nicotine					
5-Nitroacenaphthene					
2-Nitroaniline					
3-Nitroaniline.					
4-Nitroaniline.					
5-Nitro-o-anisidine					
4-Nitrobiphenyl					
Nitrofen					
5-Nitro-o-toluidine					
Nitroquinoline-1-oxide					
N-Nitrosodi-n-butylamine					
N-Nitrosodiethylamine					
N-Nitrosodimethylamine					
N-Nitrosodiphenylamine					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
N-Nitrosomethylethylamine					
N-Nitrosomethylphenylamine					
N-Nitrosomorpholine					
N-Nitrosopiperidine					
N-Nitrosopyrrolidine					
trans-Nonachlor					
Norflurazon					
2,2′,3,3′,4,5′,6,6′-Octachlorobiphenyl					
Octamethyl pyrophosphoramide					
4,4'-Oxydianiline					
Parathion					
PCB–1016					
PCB–1221					
PCB–1232					
PCB–1242					
PCB–1248					
PCB–1254					
PCB–1260	19-130	77	19-130	D-164	128
PCB–1268					
Pebulate					
Pentachlorobenzene					
Pentachloronitrobenzene					
2,2′,3,4′,6-Pentachlorobiphenyl					
Pentachloroethane					
Pentamethylbenzene					
Perylene					
Phenacetin					
cis-Permethrin					
trans-Permethrin					
Phenobarbital					
Phenothiazene					
1,4-Phenylenediamine					
1-Phenylnaphthalene					
2-Phenylnaphthalene					
Phorate					
Phosalone					
Phosmet					
Phosphamidon					
Phthalic anhydride					
alpha-Picoline (2-Methylpyridine)					
Piperonyl sulfoxide					
Prometon					
Prometryn					
Pronamide					
Propachlor					
Propazine					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Propylthiouracil					
Pyridine					
Resorcinol (1,3-Benzenediol)					
Safrole					
Simazine					
Simetryn					
Squalene					
Stirofos					
Strychnine					
Styrene 9					
Sulfallate					
Tebuthiuron					
Terbacil..					
Terbufos					
Terbutryn					
alpha-Terpineol					
1,2,4,5-Tetrachlorobenzene					
2,2′,4,4′-Tetrachlorobiphenyl					
2,3,7,8-Tetrachlorodibenzo-p-dioxin					
2,3,4,6-Tetrachlorophenol					
Tetrachlorvinphos					
Tetraethyl dithiopyrophosphate					
Tetraethyl pyrophosphate					
Thianaphthene (2,3-Benzothiophene)					
Thioacetamide					
Thionazin					
Thiophenol (Benzenethiol)					
Thioxanthone					
Toluene-1,3-diisocyanate					
Toluene-2,4-diisocyanate					
o-Toluidine					
Toxaphene 3,5					
Triadimefon					
1,2,3-Trichlorobenzene					
2,4,5-Trichlorobiphenyl					
2,3,6-Trichlorophenol					
2,4,5-Trichlorophenol					
Tricyclazole					
Trifluralin					
1,2,3-Trimethoxybenzene					
2,4,5-Trimethylaniline					
Trimethyl phosphate					
Triphenylene					
Tripropyleneglycolmethyl ether					
1,3,5-Trinitrobenzene					

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Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P1, P2 (%)	Limit for RPD (%)
Tris(2,3-dibromopropyl) phosphate					
Tri-p-tolyl phosphate					
O,O,O-Triethyl phosphorothioate.					
Trithiane					
Vernolate					

Many of the analytes in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these analytes, acceptance criteria must be developed and applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA–821–B–98–003) March 1999). Alternatively, analytes that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.

* At a minimum, these compounds must be spiked into the MS/MSD analyses when direction cannot be obtained from the data user.

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Attachment VIII – Method 625.1 Suggested Internal Standards and Surrogates

Compound	Range for Surrogate Recovery	
	Calibration Verification	Recovery from Samples
Base/Neutral Fraction		
Acenaphthalene-d_8	66-152	33-168
Acenaphthene-d_{10}	71-141	30-180
Aniline-d5		
Anthracene-d_{10}	58-171	53-142
Benzo(a)anthracene-d_{12}	28-357	22-329
Benzo(a)pyrene-d_{12}	32-194	32-194
4-Chloroaniline-d_4	1-145	1-145
bis(2-Chloroethyl)ether-d_8	52-194	25-222
Chrysene-d_{12}	23-290	23-290
Decafluorobiphenyl		
4,4'-Dibromobiphenyl		
4,4'-Dibromooctafluorobiphenyl		
1,4-Dichlorobenzene-d_4	65-153	11-245
2,2'-Difluorobiphenyl		
Dimethyl phthalate-d_6	47-211	1-500
Fluoranthene-d_{10}	61-164	38-172
4-Fluoroaniline		
1-Fluoronaphthalene		
2-Fluoronaphthalene		
2-Methylnaphthalene-d_{10}	50-150	50-150
Naphthalene-d_8	71-141	22-192
Nitrobenzene-d_5	46-219	15-314
2,3,4,5,6-Pentafluorobiphenyl		
Perylene-d_{12}		
Phenanthrene-d_{10}	67-149	34-168
Pyrene-d_{10}	48-210	28-196
Pyridine-d_5		
Acid Fraction		
2-Chlorophenol-d_4	55-180	33-180
2,4-Dichlorophenol-d_3	64-157	34-182
4,6-Dinitro-2-methylphenol-d_2	56-177	22-307
2-Fluorophenol		
4-Methylphenol-d_8	25-111	25-111
2-Nitrophenol-d_4	61-163	37-163
4-Nitrophenol-d_4	35-287	6-500
Pentafluorophenol		
2-Perfluoromethylphenol		
Phenol-d_5	48-208	8-424

Many of the surrogates in this table do not have QC acceptance criteria. If calibration is to be verified and other QC tests are to be performed for these surrogates, acceptance criteria must be developed and

applied. EPA has provided guidance for development of QC acceptance criteria (see 40 CFR 136.6(b)(2)(i) and *Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water* (EPA–821–B–98–003) March 1999). Alternatively, surrogates that do not have acceptance criteria may be based on laboratory control charts, or 60 to 140% may be used.

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Attachment IX – DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity

Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseat compression fitting
No calibration gas peaks	Cal gas valve not open	Open cal gas valve
	Calibration gas solenoid valve stuck open. All calibration gas evaporated.	Have solenoid replaced. Put fresh PFBTA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	"Blown" septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.

4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification

records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.7 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the

MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 through 6) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 through 6, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 through 6) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.20 The method blank shall be considered to be contaminated if:

- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
- If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	1645	78.5	13	40	117
95-94-3	1,2,4,5-Tetrachlorobenzene	1810	77.8	13.7	37	119
120-82-1	1,2,4-Trichlorobenzene	3577	75.7	13.9	34	118
95-50-1	1,2-Dichlorobenzene	3352	74.6	14	33	117
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	203	79.4	11.9	44	115
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2039	83	13.9	41	125
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	154	89.2	10.7	57	121
541-73-1	1,3-Dichlorobenzene	3288	72.6	14.1	30	115
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	598	84.6	14	43	127
106-46-7	1,4-Dichlorobenzene	3793	73.1	13.9	31	115
100-25-4	1,4-Dinitrobenzene	248	84.4	15.7	37	132
130-15-4	1,4-Naphthoquinone	150	81.2	8.8	55	108
90-13-1	1-Chloronaphthalene	119	81.1	11.1	48	115
90-12-0	1-Methylnaphthalene	3004	79.2	13.2	40	119
58-90-2	2,3,4,6-Tetrachlorophenol	1724	84.7	13.6	44	125
935-95-5	2,3,5,6-Tetrachlorophenol	227	75.9	11.9	40	112
608-27-5	2,3-Dichloroaniline	108	82.4	13	44	121
95-95-4	2,4,5-Trichlorophenol	4014	82.6	13.7	41	124
118-79-6	2,4,6-Tribromophenol	2930	85.7	15.4	39	132
88-06-2	2,4,6-Trichlorophenol	4183	82.1	14.5	39	126
120-83-2	2,4-Dichlorophenol	3794	80.9	13.7	40	122
105-67-9	2,4-Dimethylphenol	3886	78.4	16.2	30	127
121-14-2	2,4-Dinitrotoluene	4075	86.8	12.9	48	126
87-65-0	2,6-Dichlorophenol	1364	79.2	12.6	41	117
606-20-2	2,6-Dinitrotoluene	3706	85	13	46	124
53-96-3	2-Acetylaminofluorene	175	94	13.3	54	134
91-58-7	2-Chloronaphthalene	3569	77.5	12.1	41	114
95-57-8	2-Chlorophenol	3977	77.3	14.5	34	121
321-60-8	2-Fluorobiphenyl	3191	79.5	11.8	44	115
367-12-4	2-Fluorophenol	3008	75.2	13.3	35	115
91-57-6	2-Methylnaphthalene	5059	80.1	14	38	122
95-48-7	2-Methylphenol (o-Cresol)	4016	77	14.9	32	122
88-74-4	2-Nitroaniline	3639	85.4	13.8	44	127
119-75-5	2-Nitrodiphenylamine	279	88.1	11.6	53	123
88-75-5	2-Nitrophenol	3804	79.6	14.5	36	123
109-06-8	2-Picoline [2-Methylpyridine]	181	64.5	12.7	27	103
91-94-1	3,3'-Dichlorobenzidine	3521	71.3	16.5	22	121
56-49-5	3-Methylcholanthrene	188	95.1	13	56	134
99-09-2	3-Nitroaniline	3454	75.9	14.3	33	119
65794-96-9	3/4-Methylphenol [m/p-Cresol]	2900	76.5	14.1	34	119
534-52-1	4,6-Dinitro-2-methylphenol	3739	80.7	17.2	29	132
101-55-3	4-Bromophenyl phenyl ether	3708	85.1	13	46	124
59-50-7	4-Chloro-3-methylphenol	3880	83.3	12.9	45	122

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CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
106-47-8	4-Chloroaniline [p-Chloroanlinie]	3435	61.3	14.9	17	106
7005-72-3	4-Chlorophenyl phenyl ether	3673	83	12.7	45	121
106-44-5	4-Methylphenol [p-Cresol]	1555	84.1	14.1	42	126
100-02-7	4-Nitrophenol	3976	80.6	17	30	132
99-55-8	5-Nitro-o-toluidine [2-Amino-4-nitrotoluene]	187	69.8	15.8	23	117
57-97-6	7,12-Dimethylbenz(a)-anthracene	338	96.2	15.3	50	142
83-32-9	Acenaphthene	5300	81.3	13.7	40	123
208-96-8	Acenaphthylene	5194	81.8	16.8	32	132
98-86-2	Acetophenone	2101	73.9	13.6	33	115
120-12-7	Anthracene	5250	85.2	12.7	47	123
1912-24-9	Atrazine	1428	87.1	13.4	47	127
103-33-3	Azobenzene	378	82.1	14.2	39	125
56-55-3	Benz(a)anthracene	5385	87.4	12.9	49	126
50-32-8	Benzo(a)pyrene	5500	86.9	13.9	45	129
205-99-2	Benzo(b)fluoranthene	5323	88.3	14.5	45	132
191-24-2	Benzo(g,h,i)perylene	5263	88.5	15.1	43	134
207-08-9	Benzo(k)fluoranthene	5386	89.6	14.2	47	132
100-51-6	Benzyl alcohol	2895	75.7	15.6	29	122
111-91-1	bis(2-Chloroethoxy)methane	3705	78.4	14.2	36	121
111-44-4	Bis(2-chloroethyl) ether	3711	75.4	14.9	31	120
39638-32-9	bis(2-Chloroisopropyl) ether	769	82	16.3	33	131
117-81-7	Bis(2-ethylhexyl) phthalate	4018	91.9	13.7	51	133
103-23-1	bis(2-Ethylhexyl)adipate	156	90.8	10.1	61	121
85-68-7	Butyl benzyl phthalate	3956	90.3	14	48	132
105-60-2	Caprolactam	1203	81.3	11.9	46	117
86-74-8	Carbazole	3095	86.3	12	50	123
510-15-6	Chlorobenzilate	172	99.7	16.9	49	150
218-01-9	Chrysene	5395	87.1	12.2	50	124
84-74-2	Di-n-butyl phthalate	4041	89.4	12.8	51	128
117-84-0	Di-n-octyl phthalate	3985	92.4	16	45	140
2303-16-4	Diallate [cis or trans]	173	93.7	12.7	56	132
53-70-3	Dibenzo(a,h)anthracene	5393	89.5	14.7	45	134
132-64-9	Dibenzofuran	3749	81.5	12.7	44	120
84-66-2	Diethyl phthalate	4012	87.2	12.3	50	124
60-51-5	Dimethoate	137	68	13.3	28	108
131-11-3	Dimethyl phthalate	4023	85.9	12.6	48	124
60-11-7	Dimethylaminoazobenzene	177	98.7	11.6	64	134
88-85-7	Dinoseb	123	67.3	17.1	16	119
101-84-8	Diphenyl ether	114	95.6	6	78	114
122-39-4	Diphenylamine	854	79.5	10.6	48	111
62-50-0	Ethyl methanesulfonate	174	85.1	16.9	34	136
206-44-0	Fluoranthene	5340	88.3	12.9	50	127
86-73-7	Fluorene	5150	84.2	13.8	43	125

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CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
118-74-1	Hexachlorobenzene	4138	83.5	13	45	122
87-68-3	Hexachlorobutadiene	4003	77.3	15.3	32	123
67-72-1	Hexachloroethane	4049	72.2	14.9	28	117
1888-71-7	Hexachloropropene	259	81.9	16.7	32	132
95-13-6	Indene	188	85.3	8.9	59	112
193-39-5	Indeno(1,2,3-cd)pyrene	5367	89.3	14.7	45	133
465-73-6	isodrin	167	93.8	12.8	56	132
78-59-1	Isophorone	3787	75.9	15.2	30	122
120-58-1	Isosafrole	174	89.5	15.4	43	136
66-27-3	Methyl methanesulfonate	150	77.9	13.1	38	117
100-75-4	N-Nitorosopiperidine	232	89.4	9.8	60	119
924-16-3	N-Nitrosodi-n-butylamine	236	91.7	10.8	59	124
621-64-7	N-Nitrosodi-n-propylamine	3857	78.2	13.9	36	120
55-18-5	N-nitrosodiethylamine	421	82.1	13.8	41	124
62-75-9	N-Nitrosodimethylamine	3170	71.6	16.2	23	120
86-30-6	N-Nitrosodiphenylamine	2968	82.7	14.8	38	127
10595-95-6	n-Nitrosomethylethylamine	265	78.7	14.9	34	123
59-89-2	n-Nitrosomorpholine	172	91.3	13.8	50	133
930-55-2	n-Nitrosopyrrolidine	326	85.5	13.6	45	126
91-20-3	Naphthalene	5342	78.8	14.7	35	123
98-95-3	Nitrobenzene	4103	77.8	14.7	34	122
4165-60-0	Nitrobenzene-d5	3226	79.3	14.2	37	122
56-57-5	Nitroquinoline-1-oxide	177	91.3	24.5	18	165
126-68-1	O,O,O-Triethyl phosphorothioate	138	91.6	10.8	59	124
593-45-3	Octadecane	113	87.4	14.5	44	131
608-93-5	Pentachlorobenzene	346	89.7	11.8	54	125
76-01-7	Pentachloroethane	131	70.4	10.6	39	102
87-86-5	Pentachlorophenol	4161	78.7	18	25	133
82-68-8	Pentchloronitrobenzene	579	86.1	16	38	134
62-44-2	Phenacetin	185	95	12.5	57	133
85-01-8	Phenanthrene	5259	85.4	12	50	121
108-95-2	Phenol	4029	77.3	14.4	34	121
4165-62-2	Phenol-d5	1016	77.4	14.9	33	122
23950-58-5	Pronamide	179	93	12.4	56	130
129-00-0	Pyrene	5518	87.2	13.3	47	127
91-22-5	Quinoline	219	90	11.9	54	126
94-59-7	Safrole	176	87.8	13.6	47	129
1718-51-0	Terphenyl-d14	3111	90.5	12.3	54	127
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	136	94.4	14	52	137
297-97-2	Thionazine	139	94.6	10.7	62	127

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Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	2247	82.1	11.1	49	115
95-94-3	1,2,4,5-Tetrachlorobenzene	2326	77.9	14.5	35	121
120-82-1	1,2,4-Trichlorobenzene	4716	72.6	14.5	29	116
95-50-1	1,2-Dichlorobenzene	4442	71.4	13.3	32	111
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	112	83.9	8.3	59	109
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2244	85.4	12.2	49	122
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	241	89.1	16	41	137
541-73-1	1,3-Dichlorobenzene	4375	68.6	13.6	28	110
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	601	88.2	13.1	49	128
106-46-7	1,4-Dichlorobenzene	5433	70.4	13.9	29	112
90-13-1	1-Chloronaphthalene	211	84.5	8.8	58	111
90-12-0	1-Methylnaphthalene	3742	80	13.1	41	119
134-32-7	1-Naphthylamine	258	73.7	16.6	24	124
58-90-2	2,3,4,6-Tetrachlorophenol	2293	89	13	50	128
935-95-5	2,3,5,6-Tetrachlorophenol	266	85.6	11.7	50	121
608-27-5	2,3-Dichloroaniline	150	99.2	9.8	70	129
95-95-4	2,4,5-Trichlorophenol	5707	88.1	11.8	53	123
118-79-6	2,4,6-Tribromophenol	2059	91.5	16	43	140
88-06-2	2,4,6-Trichlorophenol	6136	87.2	12.4	50	125
120-83-2	2,4-Dichlorophenol	5330	84	12.2	47	121
105-67-9	2,4-Dimethylphenol	5298	77.5	15.6	31	124
51-28-5	2,4-Dinitrophenol	5127	82.9	20	23	143
121-14-2	2,4-Dinitrotoluene	6032	92.3	11.8	57	128
87-65-0	2,6-Dichlorophenol	1583	84	11.4	50	118
606-20-2	2,6-Dinitrotoluene	5107	90.7	11.2	57	124
53-96-3	2-Acetylaminofluorene	228	98.9	12.9	60	138
91-58-7	2-Chloronaphthalene	5084	78	12.8	40	116
95-57-8	2-Chlorophenol	5571	77.5	13.2	38	117
93951-73-6	2-Chlorophenol-d4	119	79.9	8.7	54	106
321-60-8	2-Fluorobiphenyl	2263	81.2	12.4	44	119
367-12-4	2-Fluorophenol	2022	68.8	16.6	19	119
91-57-6	2-Methylnaphthalene	6330	80.7	13.6	40	121
95-48-7	2-Methylphenol (o-Cresol)	5800	73	14.5	30	117
88-74-4	2-Nitroaniline	4855	90.8	12.1	55	127
119-75-5	2-Nitrodiphenylamine	272	97.3	11.3	64	131
88-75-5	2-Nitrophenol	5097	84.6	12.7	47	123
109-06-8	2-Picoline [2-Methylpyridine]	195	71.6	12.6	34	109
91-94-1	3,3'-Dichlorobenzidine	4815	77.9	16.9	27	129
56-49-5	3-Methylcholanthrene	237	94	12.8	56	133
99-09-2	3-Nitroaniline	4808	84.4	14.5	41	128
65794-96-9	3/4-Methylphenol [m/p-Cresol]	3472	69.7	13.6	29	110
534-52-1	4,6-Dinitro-2-methylphenol	5097	90.1	15.5	44	137
101-55-3	4-Bromophenyl phenyl ether	5074	89.1	11.5	55	124

Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
59-50-7	4-Chloro-3-methylphenol	5338	85.5	11.3	52	119
106-47-8	4-Chloroaniline [p-Chloroanlinie]	4687	75.3	14	33	117
7005-72-3	4-Chlorophenyl phenyl ether	5071	86.7	11.3	53	121
106-44-5	4-Methylphenol [p-Cresol]	2798	72.5	15.8	25	120
99-55-8	5-Nitro-o-toluidine [2-amino-4-nitrotoluene]	260	82.1	14.6	38	126
57-97-6	7,12-Dimethylbenz(a)-anthracene	373	97.1	11.9	61	133
83-32-9	Acenaphthene	6952	84.5	12.3	47	122
208-96-8	Acenaphthylene	6662	85.3	14.7	41	130
98-86-2	Acetophenone	2877	82.1	12	46	118
120-12-7	Anthracene	6792	89.6	11	57	123
140-57-8	Aramite	100	82.8	16.3	34	132
1912-24-9	Atrazine	2328	92.8	16.4	44	142
103-33-3	Azobenzene	578	88.5	9.3	61	116
56-55-3	Benz(a)anthracene	6867	91.6	11.1	58	125
50-32-8	Benzo(a)pyrene	7045	90.8	12.4	54	128
205-99-2	Benzo(b)fluoranthene	6767	92	12.9	53	131
191-24-2	Benzo(g,h,i)perylene	6624	92	13.9	50	134
207-08-9	Benzo(k)fluoranthene	6803	93.2	12.1	57	129
100-51-6	Benzyl alcohol	3349	71.2	13.5	31	112
111-91-1	bis(2-Chloroethoxy)methane	5094	83.9	11.9	48	120
111-44-4	Bis(2-chloroethyl) ether	5139	80.8	12.6	43	118
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
67-72-1	Hexachloroethane	5904	68	15.7	21	115
95-13-6	Indene	253	93.8	13.7	53	135
193-39-5	Indeno(1,2,3-cd)pyrene	6880	92.6	13.6	52	134
465-73-6	isodrin	212	97.6	10	68	128
78-59-1	Isophorone	5190	83.3	13.7	42	124
120-58-1	Isosafrole	230	91.1	11.8	56	126
66-27-3	Methyl methanesulfonate	237	70.1	12.3	33	107
298-00-0	Methyl parathion	121	101.6	19	45	159
100-75-4	N-Nitorosopiperidine	299	88.6	10.8	56	121
924-16-3	N-Nitrosodi-n-butylamine	322	90.4	10.3	60	121
621-64-7	N-Nitrosodi-n-propylamine	5145	84	11.7	49	119
55-18-5	N-nitrosodiethylamine	488	81.8	12.9	43	121
86-30-6	N-Nitrosodiphenylamine	3743	86.8	11.9	51	123
10595-95-6	n-Nitrosomethylethylamine	311	78.7	12.7	41	117
59-89-2	n-Nitrosomorpholine	214	86.2	10.3	55	117
930-55-2	n-Nitrosopyrrolidine	716	80.8	10.8	48	113
91-20-3	Naphthalene	6953	80	13.5	40	121
98-95-3	Nitrobenzene	5955	83	12.8	45	121

Table 4. LCS Control Limits – Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
4165-60-0	Nitrobenzene-d5	2223	82.1	12.6	44	120
126-68-1	O,O,O-Triethyl phosphorothioate	212	92.6	8.8	66	119
95-53-4	o-Toluidine	296	69.9	13.2	30	110
593-45-3	Octadecane	151	89	13.1	50	128
56-38-2	Parathion	152	102.6	12.3	66	140
608-93-5	Pentachlorobenzene	401	91.1	10.7	59	123
76-01-7	Pentachloroethane	139	60.9	10.4	30	92
87-86-5	Pentachlorophenol	6083	86.4	17.1	35	138
82-68-8	Pentchloronitrobenzene	618	94.5	13.4	54	135
62-44-2	Phenacetin	241	97.9	8.9	71	124
85-01-8	Phenanthrene	6822	89.6	10.2	59	120
298-02-2	Phorate	126	88.6	16.8	38	139
23950-58-5	Pronamide	249	97	10.5	65	129
129-00-0	Pyrene	7013	91.1	11.5	57	126
91-22-5	Quinoline	249	100.1	10.5	69	132
94-59-7	Safrole	233	90	9.7	61	119
1718-51-0	Terphenyl-d14	1893	91.7	13.9	50	134
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	200	96.7	11.9	61	133
297-97-2	Thionazine	196	102	10.1	72	132

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Table 5. LCS Control Limits – Method 8270 SIM Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	2267	76.6	11.3	43	111
95-95-4	2,4,5-Trichlorophenol	169	79.9	14.9	35	125
91-58-7	2-Chloronaphthalene	615	76.7	10.5	45	108
321-60-8	2-Fluorobiphenyl	1961	80.6	11.6	46	115
91-57-6	2-Methylnaphthalene	2535	76.8	12.5	39	114
83-32-9	Acenaphthene	2813	77.7	11.2	44	111
208-96-8	Acenaphthylene	2761	77.1	12.8	39	116
120-12-7	Anthracene	2812	82.1	10.7	50	114
56-55-3	Benz(a)anthracene	2827	88	11.4	54	122
50-32-8	Benzo(a)pyrene	2789	87.3	12.5	50	125
205-99-2	Benzo(b)fluoranthene	2790	90.3	12.6	53	128
191-24-2	Benzo(g,h,i)perylene	2739	87.8	13	49	127
207-08-9	Benzo(k)fluoranthene	2761	89.3	11.2	56	123
111-44-4	Bis(2-chloroethyl) ether	192	65.4	15.8	18	113
117-81-7	Bis(2-ethylhexyl) phthalate	181	108.9	13.9	67	150
85-68-7	Butyl benzyl phthalate	144	103.5	10.6	72	135
86-74-8	Carbazole	183	79.3	14.6	36	123
218-01-9	Chrysene	2812	87.5	10.2	57	118
84-74-2	Di-n-butyl phthalate	150	106.5	12.9	68	145
117-84-0	Di-n-octyl phthalate	144	105.5	16.8	55	156
53-70-3	Dibenzo(a,h)anthracene	2778	89.2	13.2	50	129
132-64-9	Dibenzofuran	282	71.9	12.2	35	108
84-66-2	Diethyl phthalate	147	99.3	10.9	67	132
131-11-3	Dimethyl phthalate	149	99.3	9.3	71	127
206-44-0	Fluoranthene	2782	87.3	10.7	55	119
86-73-7	Fluorene	2795	80.6	11.2	47	114
118-74-1	Hexachlorobenzene	201	81.9	14.2	39	125
193-39-5	Indeno(1,2,3-cd)pyrene	2812	89.6	13.5	49	130
62-75-9	N-Nitrosodimethylamine	117	90.7	10.9	58	124
91-20-3	Naphthalene	2823	74.7	12.2	38	111
4165-60-0	Nitrobenzene-d5	531	84.7	13.6	44	125
87-86-5	Pentachlorophenol	259	82.4	15.5	36	129
85-01-8	Phenanthrene	2792	80.8	10.6	49	113
129-00-0	Pyrene	2792	85.8	10.2	55	117
1718-51-0	Terphenyl-d14	1864	95.3	12.6	58	133

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Table 6. LCS Control Limits – Method 8270 SIM Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	106	77.3	7.3	56	99
90-12-0	1-Methylnaphthalene	2566	77.9	12.5	41	115
95-95-4	2,4,5-Trichlorophenol	488	84.1	13.4	44	124
118-79-6	2,4,6-Tribromophenol	164	83.7	12.7	46	122
606-20-2	2,6-Dinitrotoluene	118	67.2	15.8	20	115
91-58-7	2-Chloronaphthalene	717	72.4	12.7	34	111
321-60-8	2-Fluorobiphenyl	747	79.2	8.8	53	106
91-57-6	2-Methylnaphthalene	2984	76.5	12.6	39	114
83-32-9	Acenaphthene	3241	80.9	11.1	48	114
208-96-8	Acenaphthylene	3234	77.8	14.4	35	121
120-12-7	Anthracene	3224	85.8	11	53	119
56-55-3	Benz(a)anthracene	3277	89.3	10.1	59	120
50-32-8	Benzo(a)pyrene	3284	86.4	11.2	53	120
205-99-2	Benzo(b)fluoranthene	3248	89.7	12.3	53	126
191-24-2	Benzo(g,h,i)perylene	3178	86	14.1	44	128
207-08-9	Benzo(k)fluoranthene	3167	89.3	11.9	54	125
111-44-4	Bis(2-chloroethyl) ether	775	77.8	12.6	40	116
117-81-7	Bis(2-ethylhexyl) phthalate	275	114.1	19.6	55	173
85-68-7	Butyl benzyl phthalate	159	90.7	17.3	39	143
86-74-8	Carbazole	631	84	13.1	45	123
218-01-9	Chrysene	3215	88.3	10.4	57	120
84-74-2	Di-n-butyl phthalate	153	102.5	14.2	60	145
117-84-0	Di-n-octyl phthalate	157	103.3	19	46	160
53-70-3	Dibenzo(a,h)anthracene	3233	87.2	14.5	44	131
132-64-9	Dibenzofuran	864	77.5	14.1	35	120
84-66-2	Diethyl phthalate	142	94.5	13.5	54	135
206-44-0	Fluoranthene	3242	89.1	10.4	58	120
86-73-7	Fluorene	3232	84.1	11.3	50	118
118-74-1	Hexachlorobenzene	947	84.8	13	46	124
87-68-3	Hexachlorobutadiene	187	84.5	14.7	40	129
193-39-5	Indeno(1,2,3-cd)pyrene	3244	88.7	13.7	48	130
62-75-9	N-Nitrosodimethylamine	162	62.5	10	33	92
91-20-3	Naphthalene	3277	78.8	11.9	43	114
4165-60-0	Nitrobenzene-d5	444	83.1	9.2	55	111
87-86-5	Pentachlorophenol	808	88.4	17.6	36	141
85-01-8	Phenanthrene	3240	83.6	10.3	53	115
129-00-0	Pyrene	3252	87.1	11.3	53	121
1718-51-0	Terphenyl-d14	642	95.1	12.4	58	132

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and penta-chlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression for each analyte: r2 ≥ 0.99; **Option 3**: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL s performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times(RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.

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Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 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 and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 through 6 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 7. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 through 6 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method 8270. Tune check can be acquired as a full scan.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune. In addition to the full scan tune check, optimization for the analytes of interest is recommended.
Deuterated Monitoring Compounds (DMCs) (surrogates)	All field and QC samples.	PAH analysis: DMCs required for polyaromatic hydrocarbon (PAH) target analytes: fluoranthene-d10 and 2-methylnaphthalene-d10. Minimum RRF for PAH DMCs: 0.40. All DMCs: Requires 50-150% recovery until in-house limits can be established.	Correct problem, and then reprep and reanalyze all samples with failing DMCs if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated samples and analytes if acceptance criteria are not met and explain in the Case Narrative.	For non-PAH target analytes, other DMCs with similar chemistry must be assigned. Laboratories may use the same extract for full scan and SIM analysis if the SIM-specific DMCs are added prior to extraction.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Performance Checks	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until the performance checks are within criteria. DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan.
Initial Calibration (ICAL) for all analytes	At instrument set-up, prior to sample analysis.	Each analyte must meet one of the following options: RSD for each analyte ≤ 20% [If pentachlorophenol is a target analyte, an RSD of ≤ 40% allowed] Or Linear least squares regression for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels required for ICAL with one calibration point at the same concentration as the daily CCV. No samples shall be analyzed until ICAL has passed.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV.	Correct problem, then rerun ICAL.	NA.	RRTs shall be compared with the most recently updated RRTs. Characteristic ions must maximize in the same scan or within one scan of each other. After any maintenance is performed which could affect retention times, RRTs may be updated based on the daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value. If pentachlorophenol is a target analyte, a %D from the true value of ±50% is allowed.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within ± 20% of true value. If pentachlorophenol is a target analyte, a %D from true value of ± 50% is allowed. All reported analytes within ±50% for end of analytical batch within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV. Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, Standards, blanks, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	Internal Standard is spiked no greater than0.40 ng/µL concentration. According to the EPA Contract Laboratory Program Statement of Work (CLP SOW), this is the concentration of internal standard specified for SIM analysis. The SOW indicates calibration standards range from 0.10 to 1.0 ng/µL, so 0.40 ng/µL is mid-range. 1, 4-dichlorobenzene-d4 is ignored for SIM
Method Blank (MB)	One per preparation batch, prior to analysis of any field samples.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch.	Laboratories may use the same extract for full scan and SIM analysis provided the applicable DMCs and IS are spiked at the appropriate concentrations. Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 8. Quality Control Requirements – Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparation batch.	A laboratory must use Table 3 through Table 6 Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch.	Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparation batch.	A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 40% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	The MSD must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. All data must be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Characteristic ions for MS confirmation	Minimum 3 ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities must be > 0. Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion.	No data can be reported without MS confirmation.	NA.	Need 3 structurally significant ions that are logical fragments – not isotopic clusters. Internal standard and DMC can use fewer than 3 ions.

Document Information

Document Number: ENV-SOP-MTJL-0085 **Revision:** 04

Document Title: Diesel Range Organics/Total Petroleum Hydrocarbons (C10 to C28) by Gas Chromatography With #2 Diesel Fuel (EPA Methods 8015B/C/D)

Department(s): SVOA

Date Information

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Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

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ENV-SOP-MTJL-0085

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Fallon Labeots (006500)	Quality Analyst 1	06 Apr 2021, 02:58:46 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Shakir Wani (010007)	Manager	30 Mar 2021, 08:52:37 AM	Approved

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STANDARD OPERATING PROCEDURE
TITLE: Diesel Range Organics/Total Petroleum Hydrocarbons (C10 to C28) by Gas Chromatography With #2 Diesel Fuel (EPA Methods 8015B/C/D)
ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 SCOPE AND APPLICATION

1.1 The diesel range organics (DRO) method is designed to determine the concentration of diesel range organics in water and soil using calibration performed with #2 diesel fuel standards. The "diesel range" corresponds to an alkane range of C_{10}-C_{28} and includes mid-range petroleum products such as diesel or fuel oil. Reporting limits are based on 100ug/mL of diesel in the extract and are as follows:

State Specific	Pace Product	Range(s)/ Markers	Quant Peaks
8015B/C/D	DRO	C_{10}-C_{28}	All between markers
DRO California LUFT	DROCA	C_{12}-C_{22}	All between markers
DRO Indiana	DROIN	C_8-C_{28}	All between markers
Wyoming	DROWY	C10-C32	All between markers

Reporting Limits (RL)

Ground water & Wastewater	0.10 mg/L
Soil & Sediment	4.0 mg/Kg
Waste (TCLP) (100→1)	1.0 mg/L

1.2 Dilutions are performed as necessary to place sample quantitation within the linear range of the calibration curve. This is equivalent to a range from 100µg/mL to 10,000µg/mL of diesel fuel in the extract.

1.3 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ENV-SOP-MTJL-0016. Updated MDL records are filed and stored in a central location within the department.

1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL) and Limits of Detection (LOD)*. Should the procedure be utilized for DoD support; then the frequency of these studies must meet the requirements of the current DoD QSM.

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2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Either one liter of water is extracted using EPA 3510, 100mL of water using EPA 3510 Reduced Volume (RV), 40mL of water using Large Volume Injection (LVI) by EPA 3511, or 15 grams of soil using EPA 3546 (microwave) is spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated as needed to meet client specific reporting limits. One to 50µL of the extract is injected into a capillary column gas chromatograph equipped with a flame ionization detector (FID). Quantitation is performed by comparing the total chromatographic area to the response of a diesel standard. All chromatographic peaks eluting within the retention time windows determined by the appropriate carbon range will be considered. The required standard for this method is #2 diesel fuel. If bulk product is available from the sampling site, a calibration curve can be analyzed using the site-specific reference.

2.2 See the current Quality Assurance Manual for equations associated with common calculations.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving

personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Water samples are collected in a 1 Liter amber bottle with Teflon™ lined caps for traditional EPA 3510C extractions or in a 100mL amber bottle with Teflon™ lined caps for 3510RV extraction. LVI samples are collected in 40mL amber vials with Teflon™ lined caps. The holding time for water samples begins at the time of collection and ends with extraction that must be performed within seven (7) days. Some methods allow extension of holding time to 14 days if preserved <2pH. Soils are collected in a glass jar with a Teflon! lined cap. The samples are stored at 4 ± 2°C from the time of collection until the time of extraction. Extraction must be performed on soils within 14 days. All analyses must take place within 40 days of extraction.

4.3 PRESERVATION AND HOLDING TIMES

Aqueous samples should be preserved to a pH of less than 2 with sodium bisulfate, hydrochloric acid, or sulfuric acid. The type of preservative and resultant pH should be documented on the field chain of custody documentation. If the aqueous sample is preserved to a pH of less than 2, the holding time is 14 days from collection to extraction and 40 days from extraction to analysis. If the aqueous sample is not preserved, the holding time is 7 days from collection to extraction. All 8015 and state specific method holding times have been set to the maximum holding time allowed – 14 days for waters and soils.

EXCEPTIONS: Holding time for aqueous samples expires 7 days from collection for the following method / protocols

FLPRO, OA2, MADEP, EPHTN, DROWM

No test can be extended past the default holding time listed.

For all aqueous samples, pH must be taken and documented to represent the sample pH at the time of extraction. If pH **>2** before the default holding time has expired, additional evaluation for proper qualification is required.

- Extracted within 7 days of collection = NO qualification
- Extracted after 7 days of collection = G1 qualification

STATE NOTE: holding time qualification is not impacted by pH of the sample at the time of extraction for the following state methods / protocols

EPHTN, FLPRO, EPHCT, DROWM, AK102/103, TPHKS, OA2, MADEP

Any sample extracted past the listed holding time should be qualified regardless of pH

- If logged with less than half the holding time remaining = T8
- All other reasons = Q

Below is a list of petroleum tests that require T2 qualification for *preservation* if sample pH>2 at the time of extraction.

FLPRO, EPHCT, DROWM, AK102/103, TPHKS, MADEP

4.4 Sample extracts shall be stored in appropriately sized vials with Teflon! lined closures (screw or crimp top) at 4 ± 2°C.

4.5 All glassware is cleaned as soon as possible after use by rinsing with the last solvent used. This is followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130°C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment. See ENV-SOP-MTJL-0057, *Glassware Cleaning*.

5.0 INTERFERENCES

5.1 Interferences can be caused by the following:

5.1.1 Contaminated solvents or reagents

5.1.2 Sample processing hardware or glassware

5.1.3 Contaminated carrier gas

5.1.4 GC parts, column surfaces or detectors

5.1.5 Sample matrix

5.1.6 Other organic compounds; including chlorinated hydrocarbons, phenols and phthalate esters are measurable.

5.2 Method interferences are reduced by washing all glassware with hot, soapy water and then rinsing it with tap water, carbon filtered water, methanol, and methylene chloride. See ENV-SOP-MTJL-0057, *Glassware Cleaning*.

5.3 High purity reagents such as pesticide grade methylene chloride must be used to minimize interference problems.

5.4 Common Chemical Contaminants:

General organic compounds: Animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, and phthalate esters are measurable under the conditions of this method. Compounds eluting within the chromatographic retention window will be included in the diesel range organic results. If excessive interferences are noted, it may be necessary to utilize extract clean-up procedures such as those specified in SW-846.

Phthalate Esters: Special precautions must be taken to avoid contamination by phthalate esters. Phthalate esters are common plasticizers, frequently found in lab ware and supplies. Some of the phthalate peaks will fall within the retention window and be included in the quantitation of the diesel range organics. Care must be exercised to minimize the presence of phthalates by avoiding the use of plastics wherever possible.

High molecular weight compounds: Samples containing high molecular weight compounds may cause residual instrument contamination. A solvent blank (injection of pure solvent) should be analyzed after such a sample to ensure that the chromatograph system is free from interferences before proceeding with additional sample analyses. To reduce carryover, the chromatography column may also require an extended bake-out to remove the high molecular weight material.

5.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for cross-contamination. Samples suspect of carryover are also re-injected for confirmation.

6.0 EQUIPMENT AND SUPPLIES

6.1 Syringes: (Use the following brands or equivalent)

VWR Cat #	Syringe Size
60361-136	10µL
60376-230	25µL
60376-220	100µL
60376-558	1mL

6.2 Volumetric Flasks, "Class A", with ground glass stoppers, various sizes, ranging from 5mL to 100mL.

6.3 Gas Chromatography

6.3.1 Instrumentation:

- Instrument ID: SVGC #7: SVGC #21
- Model #: HP 6890 or HP7890
- Heating elements - Restek – GC Racer
- Column (type, brand, size): ZEBRON ZB-5, RTX 15m/30m x 0.25 x 0. 5 or 30m equivalent
- Detector: FID
- Gases used (grade and supplier): Air – medical, H_2 – 5.0, and/or N_2 or equivalent
- Autosampler Syringes used (brand, size, type): Agilent 10µL up to 250µL
- Temperature programs can be found in each instruments maintenance log or Cyberlab.

The QC department maintains information regarding MDL studies on each instrument.

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree*

Operation. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected (unless otherwise noted).

7.2 Reagent water: Carbon filtered de-ionized water

7.3 Methylene Chloride: Burdick and Jackson Pesticide grade - VWR™ catalog number BJ300-4 or equivalent

7.4 Stock Standard Solution: Pre-made NSI environmental UST145TP 50000µg/mL or equivalent. See table below for calibration standards.

7.4.1 For 1L waters and Concentrated Soils: Prepare an intermediate standard at 10,000ppm for use in diluting calibration standards. Dilute 1mL of Stock Diesel Fuel #2 to a total of 5mL of methylene chloride. The final concentration is 10,000µg/mL.

Calibration Curve Standard Preparation in Methylene Chloride

Conc. Diesel (ppm)/ OTP (ppm)	Volume (µL) of Diesel Intermediate (10,000ppm)	Volume (µL) of OTP Intermediate (1000ppm)	Final Volume (mL)
100/5	10	5	1.0
200/10	20	10	1.0
50020	50	20	1.0
1000/25	100	25	1.0
2000/40	200	40	1.0
3000	300	-	1.0
5000	500	-	1.0
10000	1000	-	1.0

Calibration concentrations and preparation are subject to change to better meet lab and or client needs.

7.4.2 For LVI or RV and non-concentrated waters/soils, prepare an intermediate standard at 200ppm for use in diluting calibration standards. Dilute 40µL of stock Diesel Fuel #2 to a total of 10mL of methylene chloride.

Calibration Curve Standard Preparation in Methylene Chloride (LVI CALIBRATION CURVE)

Conc. Diesel (ppm)/ OTP (ppm)	Volume (µL) of Diesel Intermediate (200ppm)	Volume (µL) of OTP Intermediate (20ppm)	Final Volume (mL)
2/0.1	10	5	1.0
4/0.2	20	10	1.0
10/0.4	50	20	1.0
20/0.5	100	25	1.0
40/0.8	200	40	1.0
60	300	-	1.0
100	500	-	1.0
200	1000	-	1.0

Calibration concentrations and preparation are subject to change to better meet lab and or client needs.

7.5 Custom DROPORT Standard Solution: Pre-made NSI environmental Q-4028 9000µg/mL or equivalent. See table below for calibration standards.

DROPORT Calibration Curve Standard Preparation in Methylene Chloride

Conc. Of Custom DRO (ppm)/ OTP (ppm)	Volume (µL) of Custom DRO (9000 ppm)	Volume (µL) of OTP Intermediate (1000 ppm)	Final Volume (mL)
90/5	10	5	1.0
180/10	20	10	1.0
450/20	50	20	1.0
900/40	100	40	1.0
1800/50	200	50	1.0
4500	500	-	1.0
9000	1000	-	1.0

Calibration concentrations and preparation are subject to change to better meet lab and or client needs.

7.6 ICV & CCV Standard Preparation in Methylene Chloride

Conc. Diesel (ppm)	Volume (µL) from 50000 ppm	OTP Volume (µL) from 1000 ppm	Final Cal Std Volume (mL)
3000	**600**	**20**	**10.0**

LVI Conc. Diesel (ppm)	Volume (µL) from 3000ppm DRO CCV	OTP Volume (µL) from 1000 ppm	Final Cal Std Volume (mL)
60ppm	200	-	10.0

Conc. DROPORT Diesel (ppm)	Volume (µL) from 9000 ppm	OTP Volume (µL) from 10000 ppm	Final Cal Std Volume (mL)
1800	200	50	1.0

Concentration levels are subject to change depending on instrument with the exception of the low and high concentrations.

7.7 Stock Laboratory Control Sample/SSCV: 7,500µg/mL diesel #2. Working solution is made up at 1,500µg/mL in methylene chloride. Transfer the stock standard solution into a Teflon™-sealed screw cap bottle. Store away from light with minimal headspace at -10°C to -20°C.

7.8 Surrogate Standard: Ortho-Terphenyl (OTP), Ultra Scientific IST-480 (or equivalent), 10000ug/mL in methylene chloride. Store refrigerated. Do not store above 35□C. Surrogates are added to the field samples during the extraction process.

7.8.1 Surrogate (OTP) Intermediate standard: Dilute 0.5mL of Stock OTP to a total of 5mL of methylene chloride. The final concentration is 1,000µg/mL.

7.8.2 LVI/RV Surrogate (OTP) Intermediate standard: Dilute 20µL of Stock OTP (10,000 ppm) up to 10mL with $MeCl_2$, or equivalent. The final concentration is 20µg/mL.

7.9 **Carbon Number Locator Compounds** – the regulatory state sets quantitation ranges based on carbon number range. This solution is used to determine the beginning locator retention time and the ending locator retention time for each state specific range.

Supplier/Concentration	Dilution	Final Conc.
FL PRO Component Standard NSI C-443TP or equivalent 34,000µg/mL total (17 components at 2000µg/mL each)	Dilute 1mL of stock to 10mLs with Methylene chloride.	3400 ppm (Total)
LVI = 3400ppm Total	200µl in 10ml methylene chloride	68PPM (total)

7.10 Carbon Number Marker Compounds – the regulatory state sets quantitation ranges based on carbon number. Table 7.9 lists the compounds used as marker compounds for various carbon number ranges. The marker compounds standard is purchased from Ultra Scientific, catalog number SFL-601.

Table 7.9: Carbon Number Marker Compounds

Carbon Number	Compound	Approximate Retention Time (min.)[1]
8	n-octane	0.37
10	n-Decane	0.83
12	n-Dodecane	1.25
14	n-Tetradecane	1.63
16	n-Hexadecane	1.99
18	n-Octadecane	2.33
Surrogate	o-terphenyl	2.45
20	n-Eicosane	2.64
22	n-Docosane	2.93
24	n-Tetracosane	3.19
26	n-Hexacosane	3.45
28	n-Octacosane	3.71
30	n-Triacontane	3.95
32	n-Dotricontane	4.19
34	n-Tetratricontane	4.42
36	n-Hexatriacontane	4.65
38	n-Octatriacontane	4.85

Pace Analytical®
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STANDARD OPERATING PROCEDURE
TITLE: Diesel Range Organics/Total Petroleum Hydrocarbons (C10 to C28) by Gas Chromatography With #2 Diesel Fuel (EPA Methods 8015B/C/D)
ISSUER: Pace National – Mt. Juliet, Tennessee

Carbon Number	Compound	Approximate Retention Time (min.)[1]
40	n-Tetracontane	5.05

[1]*Approximate retention times. Actual retention times are verified during instrument calibration. These RT's are subject to change and are here as a representation only. These RT's will be different from instrument to instrument.*

8.0 PROCEDURE

NOTE: Make sure that all sample information (volume, weight, batch QC, solvent lot(s), LCS/LCSD Lot#, MS/MSD Lot#, and Surrogate Lot #) is recorded in the PrepData program.

NOTE: Waters are extracted according to ENV-SOP-MTJL-0116, *Separatory Funnel Liquid-Liquid Extraction (EPA Method 3510)*. Soil samples are extracted using ENV-SOP-MTJL-0118, *Microwave Extraction (EPA Method 3546)*. Large volume injection samples are extracted according to ENV-SOP-MTJL-0122, *Micro-Extraction Procedure for Diesel Range Organics* (EPA Method 3511). Reduced volume injection samples are extracted according to ENV-SOP-MTJL-0114, Reduced Volume Separatory Funnel Liquid-Liquid Extraction (EPA Methods 3510C, 625, 608, FL PRO). Refer to the specific SOP for additional direction regarding sample preparation.

8.1 Multiphasic Samples – CONSULT SUPERVISOR

Choice of the procedure for separating multiphase samples is highly dependent on the objective of the analysis. With a sample in which some of the phases tend to separate rapidly, the percent weight or volume of each phase is calculated and each phase is individually analyzed for the required analytes.

An alternate approach is to obtain a homogeneous sample and attempt a single analysis on the combination of phases. This approach gives no information on the abundance of the analytes in the individual phases other than what can be implied by solubility.

A third alternative is to select phases of interest and to analyze only those selected phases. This tactic must be consistent with the sampling/analysis objectives or it yields insufficient information for the time and resources expended.

8.2 Gas Chromatography

Before starting an analytical sequence, an initial check and maintenance of the instrument must be performed

8.2.1 Recommended conditions: Check Cyberlab/Maintenance logs for individual instrument conditions. Each instrument has optimum operating conditions and programs that are established for each method.

8.3 Calibration

8.3.1 Blanks - Before beginning calibration or analysis, run at least one (1) methylene chloride blank to ensure that the instrument does not have any contamination from previous use. If contamination is observed, run additional methylene chloride blanks to clean and verify that the analytical system is ready to use in field sample analysis.

8.3.2 Locator Standard - Before any new calibration curve is analyzed, a marker standard comprising of the appropriate markers (see Table 7.9) for the specific analysis requested is analyzed to set calibration range and retention times.

8.3.3 Initial Calibration - Load the autosampler with the calibration standards. Calibrate the GC with an initial eight-point calibration using the commercial diesel standard (Section 7.4). Tabulate the area response of the diesel standard. The ratio of the peak response to the amount injected, defined as the calibration factor (CF), can be calculated for the standard at each concentration. If the percent relative standard deviation (%RSD) is <20% over the working range of all standards, linearity through the origin can be assumed and the calibration factor can be used in place of a calibration curve. If the %RSD is beyond 20%, then linear calibration may be employed. When used, the correlation coefficient for linear calibration must be 0.990 or better (USACE requires 0.995 or better). See section 9.1 and 9.2 for calculations.

8.3.4 Integration - For each standard analyzed, set the data system to begin integrating peaks after the solvent front and 0.05 minutes before the apex of the C_{10} peak. Stop the integration after upper limit of the retention time of the C_{28} peak. The peak integration parameters should be set to integrate to the baseline such that the area in the unresolved complex is included. Valley-to-valley integration is not permitted.

STATE NOTE: For DRO- CA the integration should begin at 0.05min before the apex of the C_{12} peak to 0.05min and after the apex of the C_{22} peak

STATE NOTE: For DRO- IN the integration should begin at 0.05min before the apex of the C_8 peak to 0.05min and after the apex of the C_{28} peak

STATE NOTE: For DRO- WY the integration should begin at 0.05min before the apex of the C_{10} peak to 0.05min and after the apex of the C_{32} peak

CLIENT NOTE: For DROPORT the ranges are broken down to C_{10} - C_{20} and C_{20} - C_{28}. And further into C_{10} - C_{12}, C_{12} - C_{16}, C_{16} - C_{21}, and C_{21} - $C_{28.0}$

8.3.5 Second Source Calibration Verification (SSCV) – Following each initial calibration, a SSCV must be analyzed to ensure the accuracy of the standard solutions used to perform instrument calibration. The SSCV is analyzed at 1500ppm or 60ppm if LVI and is prepared using the commercial diesel standard (Section 7.6). If the response for this standard varies from the predicated response by more than ±20%, a new calibration curve must be prepared. See section 9.4 for the equation to calculate recovery.

8.3.6 Calibration Verification - The appropriate retention time and working calibration factor or linear calibration curve must be verified at the beginning of each work shift, by the injection of a retention time marker and a mid-point continuing calibration verification (CCV) standard. A 3,000µg/mL diesel standard or 60ug/mL for LVI diesel standard is used for this standard. See section 9.6 for the equation to calculate percent difference. If the response for this standard varies from the predicated response by more than ±15%, a new calibration curve must be prepared.

STATE NOTE: For DRO-CA, CCV criterion is ±10% for the petroleum range.

STATE NOTE: For DRO-WY, CCV criterion is ±20% for the petroleum range.

8.4 Retention Time Windows:

8.4.1 Before establishing windows, be certain that the GC system is within optimum operating conditions. Make three injections of the method standard throughout the course of a 72 hour period. Serial injections over less than a 72 hour period results in retention time windows that are too tight.

8.4.2 Calculate the standard deviation of the three absolute retention times for the surrogate standard.

8.4.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

8.4.2.2 In those cases where the standard deviation for a particular analyte is zero, ±0.05 min is the retention time window.

8.4.3 Retention time windows must be calculated for the surrogate standard on each GC column and whenever a new GC column is installed. Retention time information must be recorded in the Instrument log. The instrument log must reflect the date that the retention time windows are calculated and the dates of the standards used to calculate the windows. The data is retained by the laboratory and is traceable through instrument logs.

8.5 Gas Chromatograph Analysis

Typical Batch order for loading the autosampler:

Sample/QC Type	Use
Solvent Blank (minimum of 1)	Verify system is contamination free
Retention Time Marker	Prior to calibration or calibration verification and every 12/24 hours – depending on method
Calibration standard(s)/SSCV	Initial 8-point calibration followed by a Second Source Calibration Verification.
Initial Calibration Verification (ICV)	Verify initial 8-point calibration in lieu of ICAL
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
Laboratory Control Sample(s)	Extracted laboratory blank, spiked with known amount(s) of analyte of interest
1 to 20 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.
Solvent Blank	Verify system is contamination free
1 to 20 samples	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard.

8.5.1 Samples are analyzed by GC/FID. Injection volumes are 1-2µL using the conditions established in 8.1. Injection volumes for LVI/RV are 20-50µL using the conditions established in 8.1.

8.5.2 If initial calibration has been performed, verify the retention time then the continuing acceptability and accuracy of the calibration by analysis of a mid-point (3,000µg/mL diesel standard/60µg/ml diesel standard). The mid-point standard must also be run once after every 20 samples and at the end of each sequence, or once every 12 hours (whichever is more frequent).

8.5.3 CCV Criteria - Calculate the percent difference of the calibration factor from the mean calibration factor. If the calibration factors have a percent difference of >15%, the instrument must be re-calibrated.

STATE NOTE: For DRO-CA, CCV criterion is +10% for the petroleum range.

STATE NOTE: For DRO-WY, CCV criterion is +20% for the petroleum range.

8.5.4 Baseline Subtraction - A methylene chloride blank must be run every /20 samples (or every 12 hours, whichever comes first) after a CCV to determine the area generated by normal baseline bleed under the conditions prevailing in the 24 hour period. This area is generated by projecting a horizontal baseline between the retention times observed for the specified range. This area is subtracted from the DRO area generated in the same manner for the samples. Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover.

8.5.5 If any field sample extract concentration exceeds the linear range of the instrument, the extract must be diluted and re-analyzed.

8.6 Chromatogram appearance

8.6.1 Chromatogram Interpretation - The analyst should generate a value for both diesel range organics and diesel or other products. Identification of diesel or other products is performed by comparing the retention times and patterns of the peaks in the sample chromatogram to those of the peaks in the standard chromatogram. The experience of the analyst weighs heavily in the interpretation of the chromatogram. Quantitation of the diesel range organics is based on summation of all peaks eluting between n-decane and n-octacosane in the most recent retention time marker.

Baseline Subtraction - Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a resulting rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. In order to accomplish this subtraction, a methylene chloride blank should be analyzed during each 12-hour analytical shift during which samples are analyzed for DROs. The area of this chromatogram is measured in the same fashion as is used for samples by projecting a horizontal baseline across the retention time range for DROs. This area is then subtracted from the area measured for the sample and the difference in areas is used to calculate the DRO concentration,

8.6.2 Reporting Non-Diesel Detections - Other organic compounds, including chlorinated hydrocarbons, phenols, and phthalates are measurable by this

method and will be reported as diesel range organics when they fall within the determined carbon range window. A comment may be made in the data report about the presence of non-diesel materials that appear in the diesel range.

NOTE: Although the retention time window definition (n-decane to n-octacosane) introduces a bias (55 to 75% for diesel), it improves precision and reduces interferences from non-diesel range components.

9.0 DATA ANALYSIS AND CALCULATIONS

See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 retention time marker every 12/24 hours as required by the applicable method, 1 method blank for every 12 hours or 20 samples, 1 Laboratory Control Sample (LCS), 1 Initial Calibration Verification (ICV), 1 Matrix Spike/Spike Duplicate (MS/MSD), 1 Continuing Calibration Verification (CCV) every 20samples/12hours, 1 CCV at end of run. All batch information must be maintained in the preparation documentation assigned to the department.

10.4 Retention Time Marker - The marker standard comprising of the appropriate carbon range markers (see Table 7.9) is analyzed prior to calibration or calibration verification and every 12/24 hours (per specific method) to ensure accurate quantitation for the diesel range addressed in this procedure.

10.5 Initial Calibration - Run a 5 to 8 point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. The percent relative standard deviation for the initial calibration curve must be <20%. If linear calibration is used, the correlation coefficient must be 0.990 or better (USACE requires 0.995 or better).

10.6 Surrogate Control Sample - After successful calibration, analyze a surrogate control sample. This standard is also the reagent blank sample and is analyzed with every analytical batch or sequence. The surrogate recovery should be within the established limits of 50-150% recovery and the sample should not have Diesel Range Organics above 1/2 of the reporting limit.

10.7 Second Source Calibration Verification (SSCV) – Following each initial calibration, a SSCV must be analyzed to ensure the accuracy of the standard solutions used to perform instrument calibration. The response must be within ±20% of the expected concentration.

10.8 ICV/CCV - Run a mid-point Initial Calibration Verification (ICV) using the primary source standards on a daily basis before sample analysis. Also run a CCV every 10 samples during an analytical sequence. The ICV/CCV must recovery within ±15% of the expected value. A successful CCV is required at the end of the analytical sequence. All samples must be bracketed by calibration verification standards that meet the acceptance criteria.

STATE NOTE: For DRO-CA, CCV criterion is +10% for the petroleum range.

STATE NOTE: For DRO-WY, CCV criterion is +20% for the petroleum range.

10.9 Method Blank - A Method Blank is analyzed with every batch of 20 samples. The quantitation of diesel range organics must be < MDL

10.10 LCS/LCSD - A Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) pair is run with every 20 samples. The accuracy of the LCS and LCSD must be within 50-150% and the RPD must be ≤20% or within calculated historical values.

10.11 Surrogate - Calculate the surrogate standard recovery in each sample. The accuracy of the surrogate must be within 50-150% or within calculated historical values

10.11.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

10.11.2 Low recoveries may be due to the sample matrix.

10.12 MRL – The reporting limit verification when analyzed must recover within ±50% of the target concentration for the standard.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly. The reporting limit verification (MRL) must recovery within ±40% of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed or a higher concentration standard can be analyzed. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

10.13 MS/MSD - A Matrix Spike (MS)/Matrix Spike Duplicate (MSD) pair is run with every 20 samples. The accuracy of the MS and MSD must be within 50-150% and the RPD must be ≤20% or within historically calculated values.

10.14 Any sample analyte responses that are beyond the linear range of the calibration curve must be diluted and re-analyzed.

10.15 Manual Integration – All manual integrations must comply with the requirements found in ENV-SOP-MTJL-0024, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.

10.16 Field blanks, duplicates, and additional quality control samples may be recommended for specific sampling programs. Matrix spikes should use the spike levels specified for laboratory control samples. (1500µg/mL diesel)

10.17 Control limits should be established for both precision and accuracy. Control Limits must be the average recovery ±3 standard deviations (SD). The Warning Limits must be the average recovery ±2SD. See ENV-SOP-MTJL-0017, *Generation of Control Limits*.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method.

11.2.1 The analyst should look at any sample that has quantifiable compounds and check the integration.

11.2.2 All calculations must be checked.

11.2.3 All surrogate recoveries must be checked to see if they are within limits.

11.2.4 Blanks must be clean of all interfering peaks.

11.2.5 Quality control criteria should be checked for the LCS, MS, and MSD.

11.2.6 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2.7 See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

11.2.8 See ENV-SOP-MTJL-0018, *Corrective and Preventative Action*.

11.3 Initial calibration - If the initial calibration does not meet the criteria for acceptance using calibration factors, then linear regression can be utilized, as long as the correlation coefficient meets the necessary criteria. If the linear regression criteria cannot be met, additional corrective actions are required. Standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including injection port cleaning, column clipping/replacement, FID detector/jet cleaning etc. When corrective actions have been completed, the instrument should be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.4 Method Blank – The method blank should not exhibit any detections greater than the MDL. If the blank shows any detectable amount greater than the RL, the laboratory performance is out of control and the problem must be corrected. Corrective actions may include: re-analysis once. If the failure persists, re-extract the entire batch of samples, if submitted sample volume permits.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.

- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

11.5 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is out of the acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective action must be performed. Corrective actions may include instrument maintenance or re-preparing the calibration standard. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed. Samples analyzed between the last passing calibration standard and the calibration standard that is out of control must be re-analyzed.

11.6 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - If the control does not perform within the ranges listed in Attachment II, or current Pace National control limits, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

11.7 Surrogates - If the recovery is not within limits stated in Attachment II or Pace National current control limits, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem. Re-extract and re-analyze the sample if none of the above are a problem or flag the data "J1" (surrogate high) or “J2” (surrogate low).

11.7.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

11.7.2 Low recoveries may be due to the sample matrix.

11.8 MRL – If the MRL does not meet the acceptance criteria, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit for the field samples must be elevated to the higher level verified.

11.9 Second Source Calibration Verification (SSCV) – If the SSCV does not meet the accuracy requirement, the initial calibration standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including injection port cleaning, column clipping/replacement, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.10 MS/MSD – If the spike and spike duplicate do not meet the criteria listed in section 10.13, or current Pace National quality control acceptance criteria, the sample must be flagged as possible matrix interference.

11.10.1 Spike failure that result in the use of a "J" flag followed by the appropriate number, which further explains the failure concerning high or low bias

11.11 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.11.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.

11.11.2 If the MS/MSD fails (recovery less than 50% or greater than 150% and/or RPD greater than 20%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.11.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.11.4 If the surrogate exhibits high recovery in the field samples and the target analytes in the field samples are below the reporting limit, the data may be released with a J1 qualifier indicating the high bias. If the QC samples (LCS, LCSD, MS, MSD) exhibit a high bias in the surrogate and the field samples are below the reporting limit for the target analyte, the data may be released with a J1 qualifier.

11.11.5 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J4 qualifier indicating the high bias.

11.11.6 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.11.7 Sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling*.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practices be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Pace National Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 No major method modifications have been made. Pace National has added the ability to further identify DRO responses by employing the use of specific standards to provide compound information, if the DRO response is atypical from actual diesel.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Pace National uses a GC Racer (from Restek) to assist the GC oven to have a faster ramp.

13.4 EPA 3510 RV: The reduction of the size of the field sample used in this procedure is performed in accordance with section 7.1 of the published EPA method. The reduction in volume extracted along with analysis of the resulting extract using large volume injection (up to 250µL can be injected with the LVI injection port) on each GC allows for low detection limits in line with those obtained using a 1L extraction and the 1-2µL injection. Complete method validation is performed for each determinative method prior to utilizing the reduced volume extraction. This validation is maintained by the Regulatory Affairs Department and is regularly verified using LCS/LCSD, MDL studies and DOCs.

13.5 EPA 3510 RV: Extractions are performed using solvent volumes of 6mL, then 6mL and then 6mL, to accommodate the100mL sample volume; rather than using 60mL of solvent three times. The sample volumes used in this procedure, when compared to the field sample volume being extracted, remain consistent with the ratios present in the published method.

14.0 REFERENCES

14.1 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.

14.2 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.

14.3 *Nonhalogenated Organics using GC/FID*, SW-846 Method 8015B, Revision 2, December 1996.

14.4 *Nonhalogenated Organics by Gas Chromatography*, SW-846 Method 8015C, Revision 3, February 2007.

14.5 *Nonhalogenated Organics using GC/FID*, SW-846 Method 8015D, Revision 4, June 2003.

14.6 *Separatory Funnel Liquid-Liquid Extraction*, Sw-846 Method 3510C, Revision 3, December 1996.

14.7 *Organic Compounds in Water by Microextraction*, SW-846 Method 3511, Revision 1, July 2014.

14.8 *Microwave Extraction*, SW-846 Method 3546, Revision 0, February 2007.

14.9 *Ultrasonic Extraction*, SW-846 Method 3550C, Revision 3, February 2007.

14.10 *California - Leaking Underground Fuel Tank Field Manual: Guidelines For Site Assessment, Cleanup, and Underground Storage Tank Closure*, October 1989, State of California Leaking Underground Fuel Tank Task Force.

14.11 *California - Leaking Underground Fuel Tank Field Manual: Guidelines For Site Assessment, Cleanup, and Underground Storage Tank Closure (Draft), October 2010, State of California Water Resources Control Board.*

14.12 *Risk Integrated System of Closure (RISC) Technical Resource Guidance Document*, Indiana Department of Environmental Management (IDEM), February 15, 2001, Chapter 8 “Total Petroleum Hydrocarbons”, Table 7.1.

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STANDARD OPERATING PROCEDURE
TITLE: Diesel Range Organics/Total Petroleum Hydrocarbons (C10 to C28) by Gas Chromatography With #2 Diesel Fuel (EPA Methods 8015B/C/D)
ISSUER: Pace National – Mt. Juliet, Tennessee

Attachment I: Revision History

Current Version (Pace National):

Date	Description of Revisions
3/27/2021	Technical and quality review and update. Revised header. Replaced section 3.0, Health and Safety. Added section 4.3 and renumbered as necessary.

Superseded Versions (ESC Lab Sciences SOP #330350A):

Version	Date	Description of Revisions
0	4/14/04	Origination
1	8/17/09	Technical and Quality Review and update.
2	7/5/12	Technical and Quality Review and update. Revised Attachment II and sections 2.1, 2.2, 2.5, 2.9, 4.2, 6.3.1, 7.5, 7.6, 7.7, 8.0 (note), 8.2.2, 8.2.6, 8.4, 9.1, 9.2, 9.4 through 9.6, 10.3, 10.4, 10.7, 10.9, 11.1, 11.8, 12.1 and 14.1; Added sections 1.3.1, 2.12 through 2.21, 5.5, 7.5, 10.12 through 10.15, 11.9 through 11.11, 13.4 through 13.5 and 14.2 through 14.5; Added state notes in sections 8.2.3, 8.2.4, Deleted sections 7.4 through 7.6.
3	8/13/15	Technical and Quality Review and update. Revised Sections 2.1, 2.2, 4.2, 5.5, 6.2, 6.3.1, 7.1, 7.4.1, 7.4.2, 7.6, 7.7, 7.8.2, 8.1.1, 8.2.2, 8.2.3, 8.2.5, 8.2.6, 8.4, 8.4.4, 8.5.2, 10.3, 10.8, 10.9, 10.10, 10.11, 10.12, 10.17, 11.2.8, 11.3, 11.6, 11.8, 11.11.2, 12.2, and 13.3. Added Note in Sections 8.0, 8.2.4, 8.2.6, 8.4.3, and 10.8. Deleted State Note in Sections 1.0, 8.2.3, 8.2.4, 11.4, 11.11.1, and 11.11.7. Deleted Section 8.5.3 and 14.5. Deleted Attachment II. Added Table in Section 1.1. Added Section 7.9, 8.1
4	12/11/2015	Technical and quality review and update. Header and signature bar re-formatting. Revised Sections 10.9, 11.4, 11.5, and 12.2. Added Attachment II.
5	11/17/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 2.2, 4.4, 5.2, 7.1, 7.4.1, 7.4.2, 7.5, 9.0, 11.5, and Attachment II Table 2. Deleted Sections 2.2 through 2.21 and 9.1 through 9.6.
6	11/29/2017	Technical and quality review and update. Changed ESC logo. Revised Sections 3.1, 14.1, 14.12, and Attachment II Table 5. Added Sections 14.2 through 14.9.

Superseded Versions (Pace National):

Date	Description of Revisions
12/28/2018	Technical and quality review and update. Deleted header, footer and signature bar. Revised sections 1.1, 1.2, 1.3, 1.3.1, 2.1, 3.0, 4.2, 4.4, 5.2, 7.1, 7.3, 7.4, 7.4.1, 7.4.2, 7.5, 7.7, 7.8.1, 7.8.2, 7.9, 7.10, 8.0, 8.2.1, 8.3.1, 8.5, 8.5.1, 8.5.2, 10.1, 10.2, 10.3, 10.9, 10.15, 10.16, 10.17, 11.1, 11.2, 11.2.7, 11.2.8, 11.6, 11.7, 11.10, 11.11.7, 12.1, 12.2, 13.1, 13.3, 13.4, 14.10 and 14.12. Corrected numbering in section 8.4.2. Revised Attachment I.
12/8/2019	Added corporate header and footer. Revised sections 5.2 and 8.0.

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STANDARD OPERATING PROCEDURE
TITLE: Diesel Range Organics/Total Petroleum Hydrocarbons (C10 to C28) by Gas Chromatography With #2 Diesel Fuel (EPA Methods 8015B/C/D)
ISSUER: Pace National – Mt. Juliet, Tennessee

Attachment II: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701BA Version B.01.00 or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty ECD	Clean ECD.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks; matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification

records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.6 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the

MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.19 The method blank shall be considered to be contaminated if:

- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
- If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 3. LCS Control Limits – Method 8015 (MOD) Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	1263	100.7	11.1	67	134
303-04	Diesel Range Organics (DRO)	2184	85.2	15.7	38	132
307-27	Gasoline Range Organics (GRO)	1134	100.3	7.2	79	122
307-51	Motor Oil	658	72.2	11.2	39	106
84-15-1	o-Terphenyl	314	87.4	14.1	45	130

Table 4. LCS Control Limits – Method 8015 (MOD) Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	756	101	10.8	69	133
303-04	Diesel Range Organics (DRO)	1757	83.7	16	36	132
307-27	Gasoline Range Organics (GRO)	971	99.9	7.3	78	122
307-51	Motor Oil	573	76.9	12.1	41	113
84-15-1	o-Terphenyl	299	90.5	11.4	56	125
630-02-4	Octacosane	130	101.1	13.8	60	142

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 20%; **Option 2:** linear least squares regression for each analyte: r2 ≥ 0.99; **Option 3:** non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane, and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected >1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use Table 3 and 4 limits or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

Document Information

Document Number: ENV-SOP-MTJL-0087 **Revision:** 03

Document Title: BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Methdo 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Luoisiana, AK101 GRO)

Department(s): VOA

Date Information

Effective Date: 10 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MTJL-0087 **Revision:** 03

Title: BTEX (Method 8021B, 602, SM6200C 2th) and Gasoline Range Organics (Methdo 8015B, 8015C, 8015D) by GC (with provisions for Calif-Lo, NWTPH-Gx, OA1, WI GRO (synthetic), Wyoming LAUST Req., GRO Luoisiana, AK101 GRO)

All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0087

QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality	10 Feb 2021, 12:24:28 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Operations	10 Feb 2021, 12:24:59 PM	Approved
[redacted]	Supervisor	10 Feb 2021, 12:26:04 PM	Approved

STANDARD OPERATING PROCEDURE
TITLE: BTEX GRO
ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0086.

1.1 BTEXM/GRO by gas chromatograph determines the concentration of benzene, toluene, ethylbenzene, m, p & o-xylene, MTBE, and gasoline range organics (C_6 - C_{10}) (range defined in Method 8015B) in solution. All matrices, including groundwater, aqueous samples, TCLP extracts, wastewater, soils, sludge, sediments, and other solid wastes, can be analyzed by this method. Wisconsin GRO and AK101 GRO are determined by this method. Samples analyzed by the GRO-Louisiana method are quantitated using a carbon range of C_6 – C_{10}.

1.2 The data shown in Attachment II provides the reporting limits for analytes in clean aqueous samples for each instrument currently running this method; however, reporting limits are subject to change to address matrix issues, to better meet client/project/regulatory needs or to improve laboratory method performance.

1.3 Method Detection Limits (MDLs) are performed and evaluated based on ENV-SOP-MTJL-0016. Updated MDL records are filed and stored in a central location within the department.

1.3.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ).* Should the procedure be utilized for DOD support; then the frequency of these studies must meet the requirements of the current DOD QSM.

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 Samples (except for those to be prepared by 5035A) require no preparation before analysis unless the concentration of the analyte is great enough to require a serial dilution.

In samples being analyzed by 5035A, an aliquot of the methanol extract is used to prepare the necessary dilution in 5mL of DI water.

2.2 This SOP describes the determination of concentrations of benzene, toluene, ethylbenzene, MTBE, and m, p, & o-Xylene by PID and gasoline range organics by FID. This method uses purge and trap to determine these concentrations. The BTEXM compounds and GRO concentrations are determined by internal standard calibration using fluorobenzene as the internal standard. Sample Introduction Method: The volatile compounds are introduced into the gas chromatograph by the EPA Purge-and-Trap Method 5030, SW-846.

2.2.1 Samples are placed in vials and purged with helium gas. The purged volatile compounds are transported to a trap (Supelco Purge Trap G) that is at 40! C. The trap is rapidly heated at the end of the purge cycle to 200! C and the volatile compounds desorb to the capillary column. After passing through this column, the compounds first pass by the PID, which detects double bonds, and then by the FID, which detects compounds that burn. As they pass by these detectors, an electrical signal is transmitted to a computer or integrator and causes an electrical peak to be recorded. The area underneath these peaks can be compared to known concentrations to determine the concentrations of BTEXM compounds in the sample.

2.3 Gasoline Range Organics (GROs) - Correspond to the range of alkanes from C_6 to C_{10} and covering a boiling point range of approximately 60°C – 170°C.

2.4 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical or sample being diluted in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.5 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.6 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Holding Times, Storage and Container Requirements

4.2.1 Water (EPA 5030) – See ENV-SOP-MTJL-131 for more information

Aqueous samples must be collected in triplicate in 40mL vials with the pH adjusted to <2 with HCl and stored at ≤6°C (not frozen). Sample must be analyzed within 14 days of collection. Samples not preserved with HCl must be analyzed within seven days of collection.

4.2.1.1 Samples that do not have a pH <2 are qualified with a G1 if analyzed past seven days of collection.

4.2.2 Soil (EPA 5035A) – See ENV-SOP-MTJL-0129 for more information

4.2.2.1 Sample Collection Kit – sampling kits for VOC soil samples contain at a minimum, one 2oz. jar for bulk sample collection, 2-40mL pre-weighed vials containing a magnetic stir bar, and a 5mL aliquot of preservative solution. The preservative solution in these vials is 20% sodium bisulfate solution in reagent water. Also included in the kit is a 40mL vial containing 5mL purge and trap grade methanol. The kit also contains a core sampler. Terra Core samplers are routinely used but equivalent samplers may be utilized depending on project specific needs. Samples are collected by field personnel and resealed then transported to the laboratory chilled at ≤6°C (not frozen).

4.2.2.2 Alternate kits can be provided with core samplers and a 2oz. jar for sample collection. Samples are collected by field personnel and resealed then transported to the laboratory chilled at ≤6°C (not frozen).

4.2.2.3 Sample must be analyzed within 14 days of collection. Low concentration samples that are **NOT** preserved with sodium bisulfate must be frozen or analyzed within 48 hours of collection. Upon receipt, these samples are frozen extending the holding time to 14 days.

4.2.2.4 Unprepared bulk samples coming into the laboratory are prepped as two low concentration samples and one high concentration sample if sufficient sample has been provided.

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and have a resulting pH > 12.

4.3 **STATE NOTE:** AK101 Soil/Sediment Collection Procedure - Soils and Sediments: Soil and sediment samples require special procedures to minimize the loss of volatile organic compounds during transit from the field to laboratory. **Please note that this sample preservation is different from SW-846 Method 8021B. The use of sodium bisulfate as a preservative is not acceptable.**

4.3.1 Soil or sediment samples must be collected into appropriately sized containers and submerged in surrogated methanol.

4.3.2 Solid samples must be collected with minimum disturbance into tared jars with a Teflon™-lined septum fused to the lid. Jars should be 4oz. or larger. 25mL aliquots of methanol (includes 1.25µL of a surrogate solution at 50µg/mL) are carefully added to the undisturbed soil until the sample is submerged.

4.3.3 It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution, and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented so that these weights can be calculated.

4.3.4 The ratio of soil to methanol used to calculate the MDL and PQL offered in the AK101 method was 1:1 (w:w). However, absorbent, organic soils such as muskeg and tundra require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio.

4.3.5 Soil for volatiles analysis can be collected using any coring device that minimizes soil disturbance. Any scraping, stirring, or similar activity results in a loss of volatiles during sampling. A sufficient number of samples must be collected to provide for backup in case of breakage.

4.3.6 Although it is not necessary to refrigerate all methanol preserved samples at 4° ± 2°C after collection and until analysis is complete, collected samples must be kept below 25°C.

4.3.7 A second surrogate, added to the methanol and soil mixture after sample collection, may be used in addition to, but not in place of, the surrogate with which the field methanol preservative was prepared.

4.3.8 A reagent methanol trip blank must be prepared in the same manner as the sample vials and must contain surrogated methanol. One trip blank must be included with each shipping container and must be stored and analyzed with the field samples. Trip blank analysis is not required if all samples in a shipping container are less than the project specific cleanup level.

4.3.9 Field blanks may be added to the sampling protocol and are prepared in the field by addition of surrogated methanol to the prepared container, as required by the Assessment Firm or the Project Manager.

4.3.10 A sample of the same soil to be analyzed for GRO must be collected into a moisture-proof container for per cent moisture determination. This sample is processed as soon as possible upon arrival at the laboratory to assure that the resulting moisture determination is representative of the preserved sample as surveyed.

4.3.11 Trip blanks, field blanks, method blanks, etc. are prepared from the same batch of solvent, reagents, and vials as are used for sample preservation.

4.3.12 Twenty-eight days is the maximum holding time for soil and sediment samples collected under this section.

4.3.13 Because the jars are pre-weighed, it is extremely important that the sampler put evidence tape on the kit ONLY, or the bubble bags in which the sample bottles are shipped, and not on the individual bottles. Removal of evidence tape is extremely difficult and the additional weight biases final results. Also, the glue on the evidence tape can contribute to the volatiles concentration in the sample.

4.3.14 Trip blanks, field blanks, and bottle blanks are prepared as appropriate to meet the quality assurance goals of the project plan.

4.3.15 28 days is the maximum holding time for AK101 soil samples preserved with MeOH. If BTEX is included, the holding time is 14 days.

4.4 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

5.0 INTERFERENCES

5.1 Matrix interference can result in samples with high concentrations of volatile organic compounds (VOC). This interference can cause the resulting peaks to not be clear and concise. This can lead to misidentification of compounds and/or poor quantitation of those compounds. This problem can be solved by diluting the sample.

5.2 Carryover from previous samples must be monitored through the use of sample blanks. When a sample with a high concentration of VOC's is followed by a low-level sample, false peaks may result from carryover. Sample blanks are used to clean the instrument.

6.0 EQUIPMENT AND SUPPLIES

The operation, cleaning and scheduled maintenance procedures prescribed by the equipment manufacturer are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each instrument.

6.1 **Instrumentation**

- Designated Instruments: VOCGC #1, #3, #4, #5, #6, #10, #12, #14, #15
- Use (method #'s): 8021B; 8015B, 8015C & 8015D
- Model #: HP 5890/6890 or equivalent
- Column (type, brand, size): J&W Scientific DB VRX 75m x 0.450mm, 2.55µm or equiv.
- Detector: GC FID, PID
- Software name and version: HP Chemstation G1701BA B.01.00, C.01.00 or equivalent
- Sample introduction system: Archon Autosampler, Encon P & T, or equivalent

6.2 **Glassware**

Volumetric – glassware equipped with penny head ground glass stopper. The volumetric flasks and graduated cylinders are cleaned by rinsing with methanol and laboratory reagent water. The volumetrics are dried in a low temperature oven at less than 120°C. Never use a brush or strong alkali solution to clean the volumetrics.

6.3 **Glass Sample (VOA) and Standard Vials:**

6.3.1 "42.5mL" VOA vials with a Teflon# /silicone septa and polypropylene open-top cap.

6.3.2 8mL vials with Teflon# /silicone/Teflon# septa and polypropylene open-top cap. (Used to store unused standards.)

6.3.3 2mL vials with Teflon# lined screw caps.

6.4 **Miscellaneous:**

6.4.1 Stainless Steel Spatula, tongue depressors

6.4.2 Teflon# -coated stir bars, 8mm x 16mm

6.4.3 Laboratory Blank Matrix: Sand, glass beads, etc. is prepared by rinsing clean with methanol and laboratory reagent water several times. The matrix is baked in an oven at 175ºC overnight to remove any volatiles and is then stored in the sealed container used for baking. The clean laboratory matrix may be purged with carrier grade helium or nitrogen to remove trapped volatiles.

6.5 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100!C, or equivalent

6.6 Top-loading Balance, capable of weighing to 0.01g, or equivalent

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months, or sooner, if a problem is detected unless otherwise noted.

7.2 **Reagents**

7.2.1 Nanopure water or equivalent: Nanopure water is used in all blanks to assure that it contains less than the method detection limit (MDL) of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds.

7.2.2 Methanol, CH_3OH (VWR™ #EM-MX0480-1 or equivalent) - purge and trap grade, demonstrated to be free of target analytes. Store isolated from other solvents in the designated flammables cabinet.

7.2.3 Sodium Bisulfate, $Na_2S_2O_3$ from QEC, Level 3 certified in 40mL vials, or equivalent

7.2.4 Sodium Bisulfate monohydrate, 99% for analysis, ACROS Organics or equivalent

7.3 **Stock Standards:** Stock solutions may be prepared from pure standard materials or purchased as certified solutions. These standards are prepared in methanol. Store stock standards in vials at ≤10!C.

7.3.1 BTEXM/GRO Calibration Standard – NSI PVOC/GRO mixture UST-360, or equivalent is used for the BTEXM compounds, and Restek certified BTEX in unleaded gas composite Cat # 30237, or equivalent, is used for 8015 GRO/AK101. The stock standards are prepared from standards with the following components and approximate concentrations:

Benzene	1000µg/mL
Toluene	1000µg/mL
Ethylbenzene	1000µg/mL
M & P-xylene	2000µg/mL
o-xylene	1000µg/mL
MTBE	1000µg/mL
n-Pentane	1000000µg/mL
GRO	5500µg/mL

7.3.2 Synthetic WISGRO Calibration Standard – NSI PVOC/GRO mixture UST-360, or equivalent, while the LCS is from Restek Cat # 30095 revised WISC PVOC/GRO mixture or equivalent. The stock standard is prepared from a standard with the following components and concentrations:

MTBE	1000µg/mL
Benzene	1000µg/mL
Toluene	1000µg/mL
Ethylbenzene	1000µg/mL
m-xylene	1000µg/mL
p-xylene	1000µg/mL
o-xylene	1000µg/mL
1,2,4-trimethylbenzene	1000µg/mL
GRO (sum of rep. comp.)	10000µg/mL
1,3,5-TMB	1000µg/mL
Naphthalene	1000µg/mL

7.3.3 BTEXM/GRO Laboratory Control Standard - Restek revised WISC PVOC/GRO mix cat# 30095 or equivalent for BTEXM and NSI Gas composite Q-4643 or equivalent for 8015 GRO/AK101: Concentrations as stated in 7.3.2.

7.3.4 GRO Retention Time Marker: Restek revised WISC PVOC/GRO mix cat#30095 or equivalent for BTEXM and NSI Gas composite Q4643. n-Pentane standard – Fox Scientific, Inc. pure grade or equivalent. Concentrations as stated in 7.3.1

7.4 **Intermediate ICV/CCV/LCS Standards**

7.4.1 BTEX ICV from NSI (UST-360) is certified BTEX in unleaded gas. The LCS is from Restek Cat#30095. Secondary dilution standards of BTEXM/GRO Standard: This intermediate standard is stored with minimal headspace in the same manner as the stock standard. 2.5mL of BTEXM solution from 7.3.1 in 50mL of methanol has the following concentrations. The GRO standard is not diluted.

Benzene	50µg/mL
Toluene	50µg/mL
Ethylbenzene	50µg/mL
M & P-xylene	100µg/mL
O-xylene	50µg/mL
MTBE	50µg/mL
n-Pentane	50µg/mL
GRO	5500µg/mL

(Syringe sizes needed: 5mL, 25µL, 10µL, 1 µL & 0.5 µL)

7.4.2 WISGRO's working standard is prepared by mixing 2.5mL of synthetic WISGRO standard into 47.5mL of methanol to make a 50ppm working standard which is then used to prepare all calibration standards.

7.5 **Calibration standards:**

Calibration standards are prepared in reagent water at a minimum of five concentration levels. The lowest standard must be at or below the RL. The calibration standards are prepared from the primary source (which is a different Lot # than the LCS) according to the instructions in 7.3. This is the intermediate stock. Use the measurements listed below to produce each calibration point. The concentration varies slightly with lot number.

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STANDARD OPERATING PROCEDURE
TITLE: BTEX GRO
ISSUER: Pace National – Mt. Juliet, Tennessee

BTEXM/GRO (Soil Calibration)

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	0.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	5500	0.055 µg/mL	0.11 µg/mL	0.55 µg/mL	1.1 µg/mL	2.75 µg/mL	5.5 µg/mL	11 µg/mL	-	-
GRO Surrogate - a,a,a-TFT		200µg/L	202µg/L	204µg/L	206µg/L	208µg/L	210µg/L	212µg/L	-	-
Benzene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100µg/L	200µg/L	250µg/L
Toluene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100µg/L	200µg/L	250µg/L
Ethylbenzene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100µg/L	200µg/L	250µg/L
m&p Xylene ppb	50	1µg/L	2µg/L	10µg/L	20µg/L	50µg/L	100µg/L	200µg/L	400µg/L	500µg/L
o Xylene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100µg/L	200µg/L	250µg/L
MTBE ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100µg/L	200µg/L	250µg/L
BTEX Surrogate - a,a,a-TFT		200µg/L	202µg/L	204µg/L	206µg/L	208µg/L	210µg/L	212µg/L	216µg/L	218µg/L

*Prepared in 5mLs of DI water

BTEXM/GRO (Water Calibration)

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	0.5	1	5	10	25	50	100	200	250
GRO ppm	5500	0.055 µg/mL	0.11 µg/mL	0.55 µg/mL	1.1 µg/mL	2.75 µg/mL	5.5 µg/mL	11 µg/mL	-	-
GRO Surrogate - a,a,a-TFT		200µg/L	202µg/L	204µg/L	206µg/L	208µg/L	210µg/L	212µg/L	-	-
Benzene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Toluene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Ethylbenzene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
m&p Xylene ppb	50	1µg/L	2µg/L	10µg/L	20µg/L	50µg/L	100µg/L	200µg/L	400µg/L	500µg/L
o Xylene ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
MTBE ppb	50	0.5µg/L	1µg/L	5µg/L	10µg/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
BTEX Surrogate - a,a,a-TFT		200µg/L	202µg/L	204µg/L	206µg/L	208µg/L	210µg/L	212µg/L	216µg/L	218µg/L

*Prepared in 50mLs of DI Water

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National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: BTEX GRO
ISSUER: Pace National – Mt. Juliet, Tennessee

Synthetic GRO (Soil Calibration)

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	.05	.1	.5	1	2.5	5	10	20	25
GRO ppm	500			0.05 µg/mL	0.1 µg/mL	0.25 µg/mL	0.5 µg/mL	1.0 µg/mL	2.0 µg/mL	2.5 µg/mL
Benzene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Toluene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Ethylbenzene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
m&p Xylene ppb	50	1µg/L	2ug/L	10µg/L	20ug/L	50µg/L	100ug/L	200µg/L	400ug/L	500µg/L
o Xylene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
MTBE ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L

*Prepared in 5mLs of DI water

Synthetic GRO (Water Calibration)

	Conc. ppm	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL	Amt.uL
	Stock listed in 7.3.1	.5	1	5	10	25	50	100	200	250
GRO ppm	500			0.05 µg/mL	0.1 µg/mL	0.25 µg/mL	0.5 µg/mL	1.0 µg/mL	2.0 µg/mL	2.5 µg/mL
Benzene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Toluene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
Ethylbenzene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
m&p Xylene ppb	50	1µg/L	2µg/L	10µg/L	20ug/L	50µg/L	100ug/L	200µg/L	400ug/L	500µg/L
o Xylene ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L
MTBE ppb	50	0.5µg/L	1µg/L	5µg/L	10ug/L	25µg/L	50µg/L	100ug/L	200µg/L	250µg/L

*Prepared in 50mLs of DI Water

7.6 **Internal standard Fluorobenzene** 100,000µg/mL - NSI Cat # Q-4187 or equivalent.

7.7 **Surrogate** ααα-trifluorotoluene 100,000µg/mL - NSI Cat # Q-4187 or equivalent.

7.8 **Surrogate/Internal standard preparation**: Commercially-prepared ααα-TFT at 100,000 µg/mL and Fluorobenzene at 100,000µg/mL are purchased from NSI for use in making an internal standard/surrogate mixture. This mixture is prepared by diluting 1mL of the NSI mixture into 99mL of methanol (100mL total volume). It is injected automatically by the instrument at a rate of 1µL per 5mL purge volume. This results in a 200µg/L solution of internal standard/surrogate. Check daily to make sure that the instrument reservoir has adequate IS/Surr solution.

STATE NOTE: For Wisconsin GRO/PVOC and AK101 samples, the internal standard/surrogate stock mix has Fluorobenzene at 100,000µg/mL and ααα-trifluorotoluene at 20,000µg/mL. The working standard is prepared by diluting 0.250mL of the stock mix to a final volume of 50mL using methanol.

7.9 **Solvent:** Methanol Fisher GC Resolve A457-4 or equivalent: High res.- GC grade.

7.10 **Spike Solution (LCS/MS/MSD):** For the LCS, spike the LCS spike solution prepared in section 7.3.1 into duplicate aliquots of a clean matrix. For the soil MS/MSD, prepare the

spikes using the same as LCS solution except, introduce 5µl of the solution prepared in 7.3.1 directly into separate aliquots of the selected field sample or 50µl for water matrices.

NOTE: LCSD are analyzed as required per state or agency requirements.

8.0 PROCEDURE

8.1 **Analysis Summary:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via an autosampler. If soil samples are high in contamination, a methanolic extraction may be necessary prior to purge and trap analysis. Soils require method 5035A for sample preparation, See ENV-SOP-MTJL-0129 for more information.

8.2 **Gas Chromatography Conditions:**

The particular settings for all instruments are subject to change at any time. See each individual instrument for the current autosampler, purge-and-trap, and gas chromatograph settings. Typical conditions are given in the following table:

Item	Setting
Autosampler and Purge-and-Trap	
Trap	G trap
Valve Oven Temperature	110°C
Transfer Line Temperature	110°C
Purge Time	11 minutes
Purge Flow	40mL/minute
Dry Purge Time	1 minute
Desorb Preheat Temperature	220°C
Desorb Temperature	230°C
Desorb Time	1 minute
Bake Temperature	230°C
Bake Time	2 minutes
Gas Chromatograph	
Inlet Temperature	250°C
Split Ratio	0.8:1
Column	Rtx-VRX 75m x .45mm x 2.55µm
Column Flow	10mL/minute; Hold 6 minutes
	Ramp 2mL/minute to 22mL/minute
Oven Program	45°C
	Ramp 10°C/minute to 75°C
	Hold 1 minute
	Ramp 15°C/minute to 80°C
	Ramp 30°C/minute to 230°C
	Hold 5.67 minutes
Detector Temperature	300°C

8.3 **Calibration:** Method 8015, 8021B BTEXM, WI GRO, and GROMAR require a five-point calibration curve. This curve must have a % RSD of <20% for each of the BTEXM/GRO compounds. In the event that RF criteria are not met, linear regression may be used. In order to use this option, the correlation coefficient of the calibration curve must be a minimum of 0.990 or better. Equal weighting factors or 1/x regressions may be used.

STATE NOTE: Linear regression is required for quantitation of **WI GRO** samples. PVOC is acceptable on average response.

STATE NOTE: AK101 GRO must have %RSD < 25%.

8.3.1 **Retention Time Marker:** GRO by 8015, WI GRO, and Synthetic GRO quantitation is performed using "baseline to baseline" integration. The area is summed from the marker compounds of MTBE to 1,2,4-trimethylbenzene, representing C_6 to C_{10}. These markers are evaluated, and RT's changed when appropriate. The instrument response attributed to the surrogate and internal standard is not included.

Retention Time Marker standards are analyzed at the beginning of each analytical run.

STATE NOTE: For NWTPH-Gx, the retention time range for gasoline integration must, at a minimum, include toluene through naphthalene. For surrogates that elute within the retention time range used for TPH integration, the analyst must subtract the area of the surrogate(s) from the total area of the TPH peak to yield the appropriate area of the petroleum product.

STATE NOTE: For AK101, the retention time range for gasoline integration must include the resolved and unresolved components that elute between and including C_6 (hexane) and C_9 (nonane) to end at the peak start time of C_{10} (decane). Quantitation must be performed using "baseline to baseline" integration.

8.4 **Gas chromatographic analysis:**

Typical Batch order for loading the autosampler when a calibration is run:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Also required to be analyzed every 24 hours for AK101 and GROMAR. And every 12 hours for GROWY.
Calibration standard(s)	Initial 5-point calibration or single-point calibration verification. MUST be mid-point standard.
Second Source Cal. Verification (SSCV)	Second Source verification of initial calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
Methanol Blank	Laboratory blank spiked with 200 µL laboratory grade methanol to show no contamination for methanol preserved samples
1 to 20 samples	Client samples

Typical Batch order for loading the autosampler when a calibration is not run:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
Retention Marker	Verify windows for gasoline ranges. Required to be analyzed every 24 hours for AK101 and GROMAR. And every 12 hours for GROWY.
Initial Calibration Verification (ICV)	Verify initial 5-point calibration.
Laboratory Control Sample(s)	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard, and that the analytical system does not show contamination above the established detection limits
Methanol Blank	Laboratory blank spiked with 200 µL laboratory grade methanol to show no contamination for methanol preserved samples
1 to 20 samples	Client samples

8.4.1 Water/liquid Samples

Samples are received in 40mL vials containing HCl as a preservative. The autosampler removes 5mL of sample, mixes the sample with 1µL of internal standard/surrogate mix, and heats the sample for 30 seconds at 40□C. The sample then purges for 11min at ambient temp to drive off all VOC's to the trap. The trap is desorbed for one minute at 175□C before entering the column for analysis.

8.4.2 Soil/Sediment Samples (Water Purge) - Collected in soil Jar

Weigh 5 grams of soil into a 40mL vial containing a stir bar. Add 5mL of bisulfate and tighten cap. The autosampler injects the sample with 1µl of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 1 minute at 40□C. The sample then purges for 11min at 40□C to drive off all VOC's to the trap. The trap is desorbed for one minute at 175□C before entering the column for analysis.

8.4.3 Soil/Sediment Samples - Collected in Encore (Encore "like") Sampling Device

The sample is collected using an Encore or Encore "like" sampling device. The device is designed to sample soil at approximately 5g. The sample is placed into a pre-weighed 40mL vial containing a stir bar and 5mL of Sodium Bisulfate, as a preservative. Weigh the vial to determine the weight of the soil. WISGRO is 25g of soil into 25mL of methanol.

Soil Sample Weight (g) □ Total weight of Vial and Soil (g) - Pre - weigh value (g)

Record the determined weight of the sample and load onto the autosampler. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 30 seconds at 40□C. The sample then purges for 11min at 40□C to drive off all

VOC's to the trap. The trap is desorbed for one minute at 175□C before entering the column for analysis.

8.4.4 High Level Soil/Sediment Sample (Methanol Extraction) – Collected in Soil Jar

NOTE: Samples known to have high concentrations greater than 250ppb may be collected in a 2oz. Sample jar with no headspace.

Weigh 5g of soil sample and place in vial. Add 5mL of Methanol and vortex for 30 seconds. Using a gas tight microsyringe, measure a maximum of 200µL of methanol extract and inject into a vial containing 5mL of water. Enter the sample multiplier as 25X. The autosampler injects the sample with 1µL of internal standard/surrogate mix. The autosampler moves the vial into a heating chamber and heats the sample for 30 seconds at 40□C. The sample then purges for 11min at 40□C to drive off all VOC's to the trap. The trap is desorbed for 1 minute at 175□C before entering the column for analysis. For PVOCGRO soil samples 25g of soil is placed into a vial with 25mL of methanol. If the weight of the soil exceeds 35g the sample is discarded. If the weight of the sample is >26g but <35g methanol is added until the volume of methanol in mL is equal to the weight of the soil in g.

STATE NOTE: For WI PVOC/GRO samples, a maximum of 100µL may be injected into 5mL for a multiplier of 50x. Sonicate Wisconsin soils in the methanol for 20 minutes.

8.5 **Quantitation:**

8.5.1 Quantitation of GRO is performed by the internal standard method. The concentration of Gasoline Range Organics in the sample is determined from a summation of the total responses within the ranges specified in the table below, using the calibration curve. No area other than that relating to the internal standard or surrogates may be subtracted from the GRO retention time window in calculating GRO results. WISGRO is evaluated by the external standard method. WISGRO no IS/SURR areas are subtracted and range is from the beginning of MTBE and to the conclusion of Naphthalene.

Product(s)	Range	Description
GRO, GROWY, GROAZ, GROMAR, GROMI, GROPORT, OA1	C_6-C_{10}	Peak start of MTBE to the conclusion of 1,2,4-Trimethylbenzene peak
AK101	C_6-C_{10}	Peak start of MTBE to the peak start of 1,2,4-Trimethylbenzene peak
GROWM	C_6-C_{10}	Peak start of MTBE to the conclusion of the Naphthalene peak
GROCA, GROIN	C_5-C_{12}	Peak start of Pentane to the conclusion of Naphthalene peak

Product(s)	Range	Description
GROOH, NWTPHGX	C_6-C_{12}	Peak start of MTBE to the conclusion of the Naphthalene peak

8.5.2 Integration must be "baseline to baseline" as opposed to a "valley to valley". Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of the chromatogram that includes all responses within the retention time window. The correct baseline coincides with a horizontal line drawn through the lowest point in the chromatogram before the end of the window. The lowest point may be within the window, before the window, or before the solvent front. Baseline to baseline integration does not include the solvent peak. Placement of the baseline is determined for each sample.

8.5.3 BTEX/MTBE quantitation is performed using "total area vs selected peak".

STATE NOTE: For WI PVOC/GRO samples, all peaks (and baseline rises) outside the window are to be addressed. If area outside the window is detected it must not be quantitated as part of the GRO result. Add report remark that peaks or baseline rises were detected outside the window.

8.6 For acceptance criteria and corrective actions, see sections 10.0 & 11.0.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 AK101 Moisture Correction: In order to report results for volatiles analysis of samples containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as Vt. This total solvent/water volume is calculated as follows:

$$\mu L \text{ solvent/water } V_t = \left[\frac{mL \text{ of solvent} + (\% \text{ moisture} \times g \text{ of sample})}{100}\right] \times 1000 \ \mu L/mL$$

9.2 See the current Quality Assurance Manual for equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Initial Calibration – Initial calibration curves must meet the criteria found in the following table. One concentration of the calibration standards must be at or below the RL. The remaining concentration should encompass the linear working range of the instrument.

In most cases, %RSD or linear regression is acceptable. When using linear regression, equal weighting factors or 1/x regressions may be used.

Analytical Method	Min. # of Calibration Standards Required	Initial Calibration Acceptance Criteria	
		%RSD	Linear Correlation Coefficient
EPA 8015B, 8015C, 8015D	5	≤20%	≥0.990
EPA 8021B	5	≤20%*	≥0.990*
WI PVOC	5	≤20%*	≥0.990*
WI GRO	5	NA	≥0.990
NWTPH-Gx	5	≤20%	≥0.990
EPA 602	3	≤10%	≥0.990
OA1	3	≤20%	≥0.995
AK101	3	<25%	NA
GROMAR	5	<20%	>0.990

* required for each target analyte being reported for this method.

NA indicates that this process cannot be used for this method.

NOTE: For USACE samples the correlation coefficient must be ≥0.995.

10.4 Second Source Calibration Verification (SSCV) – Initial calibration curves must be verified with a second source prior to analyzing any samples. Recoveries from a mid-point standard made from a secondary source not used to generate the calibration curve must be within ±20% of the true value before sample analysis may begin.

STATE NOTE: For samples analyzed in conjunction with AK101, recoveries from a mid-point standard made from a secondary source not used to generate the calibration curve must be within ±25%

10.5 Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) – Before beginning a sample run, a midpoint check standard (ICV) is analyzed initially to ensure accurate instrument calibration. Continuing calibration verification (CCV) must be checked after every 20 samples. Acceptance criteria for the specific methods are listed in the table below.

Analytical Method	Continuing Calibration Acceptance Criteria
EPA 8015B, 8015C, 8015D	± 20% of Expected Value
EPA 8021B	± 20% of Expected Value
WI PVOC	± 15% of Expected Value
WI GRO	± 20% of Expected Value
NWTPH-Gx	± 20% of Expected Value
EPA 602	± 30% of Expected Value
OA1	± 20% of Expected Value
AK101	± 25% of Expected Value
GROMAR	± 20% of Expected Value

10.6 Method Blank – Method Blanks contain reagent water that are analyzed following successful calibration and/or verification to ensure that the analytical system is free from interferences prior to the analysis of field samples. The acceptance criterion for all method blanks is less than the method detection limit.

NOTE: For Methanol Soils, a Methanol Blank is analyzed.

10.6.1 If more than one instrument blank or method blanks are analyzed, evaluate, and assess the blank and field samples under the same conditions for possible mid-level standard carryover using the subsequent blank after the mid-level standard on a per analyte basis.

STATE NOTE: For samples analyzed in conjunction with AK101, blank subtraction is not permitted. Blanks should be reported by value for data quality assessment.

NOTE: Additional blanks may be submitted with client batches to verify that no cross contamination occurs during shipping of samples and there is no contamination contributed from the sampling equipment. Additional blanks may also be analyzed to ensure that the analytical system remains clean following the analysis of highly contaminated samples.

10.7 Methanol Blank **–** Method blank with 200ul of Laboratory Grade Methanol add to verify reagent is free of contamination.

10.8 Matrix Spike/Matrix Spike Duplicate - are run every 20 samples when enough sample is available or requested by client. The analyst also verifies that the spikes are at the appropriate levels. Spiking levels correspond to the midpoint of the calibration curve. Acceptance criteria are available in the LIMS. If the spike recovery does not meet criteria, verify matrix interference, and apply qualifiers.

10.9 Laboratory Control Sample (LCS)– An LCS is required with each batch and evaluated using the QC limits in LIMS for BTEXM/GRO by Method 8015. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: An LCSD must be analyzed for WI samples and the LCSD must be analyzed at the conclusion of the analytical batch. WI GRO LCS must be within 80-120% for both soil and water. The RPD cannot exceed 20% for either matrix.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix. Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD.

10.10 Surrogates - must be assessed for all samples and QC in the batch. Alpha, Alpha, Alpha - TFT recovery must be within acceptance criteria listed in LIMS.

STATE NOTE: The WI PVOC Surrogate must be within >80% for both soil and water and are analyzed from the PID only.

STATE NOTE: Surrogates in AK101 must meet 60% to 120% recovery in Blanks, LCS, and LCSD. Surrogates in field samples must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in LIMS, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

STATE NOTE: Surrogates in NWTPH-Gx must meet 50-150% for both soil and water. For ease of analysis, the control limits used by the laboratory as found in LIMS, exceed the method required limits, but allow for running these samples in conjunction with other TPH analyses.

10.11 Internal standard - IS fluorobenzene, response must be within acceptance limits for all samples and quality control samples. The internal standard response must be within 50% - 200% of the response of the calibration verification standard.

10.12 Dilutions - All sample analytical results must be below the high standard of the calibration curve.

10.13 IDOC's - The analyst must demonstrate proficiency in performing the analysis as outlined in ENV-SOP-MTJL-0014, *Technical Training and Personnel Qualifications*. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made.

STATE NOTE: Wisconsin GRO requires analysis of five replicates for initial demonstration of capability. Waters must be analyzed at a concentration of 100µg/L, with recoveries falling between 80-120% of the known concentration and the RSD must be <20% to be acceptable. Soils must be analyzed at a concentration of 10mg/kg, with recoveries falling between 75-120% of the known concentration and the RSD must be <20% to be acceptable.

10.14 Retention time windows - are calculated over a 72-hr period by taking the average RT of each compound in the ICV and calculating ±3 SD from this average. This is the retention time window. Retention time windows can vary between instruments.

STATE NOTE: Wisconsin GRO requires verification of the retention time window at the beginning of each data and whenever a new GC column is installed. This can be accomplished as part of the calibration verification.

10.15 Manual Integration – All manual integrations must comply with the requirements found in ENV-SOP-MTJL-0024, *Manual Integration Procedure*. Before and after integrations must be available for review by the secondary data reviewer.

10.16 RLV – The reporting limit verification when analyzed must recover within ±50% of the target concentration for the standard. This can be assessed with the %error calculation in capture software during ICAL evaluation.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

CLIENT NOTE: For Marathon/MPC LLC/SSA samples a reporting level check standard must be analyzed after each calibration and must recover within 60-140%. If recovery of 60-140% is not met, the reporting limit must be raised, and a back calculation performed at that level. This process must be repeated until an acceptable RL recovery is achieved.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the required RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2 All data must undergo a second analyst review. The analyst checking the data must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method.

11.2.1 The analyst should look at any sample that has quantifiable compounds and check the integration.

11.2.2 All surrogate recoveries must be checked to see if they are within limits.

11.2.3 Blanks must be clean of all interfering peaks.

11.2.4 Quality control criteria should be checked for the LCS, LCSD, MS, and MSD.

11.2.5 Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2.6 See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

11.2.7 See ENV-SOP-MTJL-0018, *Corrective and Preventive Action*.

11.3 Initial calibration - If the initial calibration does not meet the criteria for acceptance using response/calibration factors, then linear regression can be utilized, as long as the correlation coefficient meets the necessary criteria. If the linear regression criteria cannot be met, additional corrective actions are required. Standards must be reviewed and re-prepared, if necessary. Instrument maintenance may also be required, including column clipping/replacement, source cleaning, etc. When corrective actions have been completed, the instrument must be re-calibrated and the acceptance criteria must be met for the analytes of interest prior to the analysis of any field samples.

11.4 Initial/Calibration Check Standard (ICV/CCV) - When the initial or continuing calibration verification is beyond the acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

11.5 Method Blank – If the method blank shows any detectable amount greater than the MDL, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective actions include re-analysis twice. If the failure persists, re-analyze the entire batch of samples, if submitted sample volume permits. Qualification of data, where the blank result is below 1/2RL is acceptable.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

11.6 Matrix Spike/Matrix Spike Duplicate - Assess that matrix spike/matrix spike duplicates were analyzed at required frequency, and that results are within acceptance criteria. Spike failure results in the use of a "J" or "V" flag. If a "J" flag is used, it is followed by the appropriate number, which further explains the failure concerning high or low response. The "V" flag is used to indicate that the sample concentration was too high to accurately evaluate the spike recovery.

11.7 Laboratory Control Sample (LCS) – A Laboratory Control Sample (LCS) is run every 20 samples. Levels correspond to the midpoint of the calibration curve.

STATE NOTE: WI GRO LCS must be within 80-120% for both soil and water. An LCS and LCSD are required with each batch. The RPD cannot exceed 20% for either matrix. Failure of the LCS results in a required of all samples within the batch.

STATE NOTE: AK101 GRO LCS must be within 60-120% for both soil and water with RPD not exceeding 20%. Surrogates in AK101 must meet 60% to 120% recovery in laboratory control samples – Blanks, LCS, and LCSD.

If the control does not perform within the ranges listed in LIMS, the laboratory performance is assumed to be out of control and the problem must be corrected. Corrective action can include re-analysis, if instrument malfunction is suspected, or re-preparation and re-analysis of the entire batch, if the failure is suspected as either extraction or sample related.

11.8 Surrogates - If the recovery is not within limits stated in LIMS, confirm that there are no errors in the calculations, surrogate solutions and standards. Check the instrument performance. Examine the chromatograms for interfering peaks and integrated areas. Re-calculate the data and/or re-analyze the field sample if any of the above checks reveal a problem. When permitted, flag the data "J1" (surrogate high) or "J2" (surrogate low).

11.8.1 High recoveries may be due to co-eluting matrix interference: examine the sample chromatogram.

11.8.2 Low recoveries may be due to the sample matrix.

STATE NOTE: The surrogate for WI PVOC must recovery >80% for both matrices.

11.9 Internal standard - The internal standard area counts must be monitored for all CCVs. ISTDs must recover within 50% to 200% of the area counts from the internal standard area counts of the midpoint standard of the most recent initial calibration sequence. If

any internal standard response is beyond the acceptable recovery, corrective action is required. Corrective action can take to form of checking the original calculations to ensure accuracy, re-analysis of the CCV to verify initial results, instrument maintenance (i.e., column clipping or changing, inlet liner cleaning/replacement, etc.) or re-calibration.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the CCV or a complete re-calibration is necessary, depending on the impact of the correction on the analytical system.

Internal standards must be monitored for each sample. ISTDs in samples must meet the –50% to +200% criteria when compared to the ISTDs in the daily CCV or mid-level of the calibration curve, on 12h shifts when full calibration is performed. Possible corrective actions include: if instrument malfunction is suspected, or re-preparation and re-analysis, if the failure is suspected as either extraction or sample related. If the sample has an obvious matrix interferent and the internal standard recovery is greater than 200%, the sample can be diluted (if acceptable reporting limits can be achieved) to minimize the interference or the sample must be re-extracted and re-analyzed to confirm the original results.

11.10 RLV - If the acceptance criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

11.11 Second Source Calibration Verification (SSCV)

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051*, Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Provisions for additional QC and specific variations have been added.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.4 With the exception of samples for WI PVOC/GRO, a maximum of 200µL of methanol extract is injected into 5mL of water for a multiplier of 25X. For WI PVOC/GRO samples, a maximum of 100µL is injected into 5mL for a multiplier of 50X.

14.0 REFERENCES

14.1 *Nonhalogenated Organics Using GC/FID*, SW-846 Method 8015B, Revision 2, December 1996.

14.2 Aromatic and Halogenated Volatiles by Gas Chromatography using Photoionization and/or Electrolytic Conductivity Detectors, SW-846 Method 8021B, Revision 2, December 1996.

14.3 *Modified GRO Method for Determining Gasoline Range Organics,* Wisconsin DNR, September 1995.

14.4 *R211 – Specific Requirements*: *Wyoming Storage Tank Remediation Testing Laboratory Accreditation Program, A2LA Document* R211, June 30, 2015.

14.5 *Method 602 – Purgeable Aromatics*, 40 CFR Part 136 Appendix A.

14.6 *Volatile Organic Compounds Purge and Trap Capillary-Column Gas Chromatographic Method*, SM 6200C, Standard Methods for the Examination of Water and Wastewater.

14.7 *Method for Determination of Volatile Petroleum Hydrocarbons (Gasoline),* Iowa Method OA-1 Revision 7/27//93, The University of Iowa, Hygienic Laboratory.

14.8 *Leaking Underground Fuel Tank Guidance Manual*, California State Water Resources Control Board, September 2012.

14.9 State of Alaska, Dept. of Env. Conservation, Contaminated Sites Laboratory Approval Memorandum, Soil Moisture Corrected Reporting by EPA Method 8000C, February 2008.

14.10 *NWTPH-Gx Volatile Petroleum Products Method for Soil and Water*, Oregon Department of Environmental Quality.

14.11 *Method AK101 for the Determination of Gasoline Range Organics*, Version 4/08/02.

14.12 *Nonhalogenated Organics by Gas Chromatography,* SW-846 Method 8015C, Revision 3, February 2007.

14.13 *Nonhalogenated Organics Using GC/FID*, SW-846 Method 8015D, Revision 4, June 2003.

14.14 *Determinative Chromatographic Separations*, SW846 Method 8000B, Revision 2, September 1996.

14.15 *Determinative Chromatographic Separations*, SW846 Method 8000C, Revision 3, March 2003.

14.16 *Louisiana Department of Environmental Quality Leaking Underground Storage Tank Program Quality Assurance Project Plan,* Louisiana DEQ, Revision 10, 5/6/2008.

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: BTEX GRO
ISSUER: Pace National – Mt. Juliet, Tennessee

Attachment I: Revision History

Current Version (Pace National):

Date	Description of Revisions
2/10/2021	Revision to change AK101 calibration standard. Revised Header as well as Sections 7.3.1 and 7.3.3.

Superseded Versions (ESC Lab Sciences SOP#330351):

Version	Date	Description of Revisions
0	8/94	Origination
1	7/95	
2	12/28/98	
3	9/1/99	
4	8/22/00	
5	11/1/01	
6	4/29/02	
7	4/23/03	
8	11/3/03	
9	4/14/04	
10	8/15/06	
11	11/30/07	Technical and Quality Review and update.
12	1/10/08	Addition of Section 4.5.4 - AK101 requirements.
13	2/23/09	Clarification of spike solutions in section 7.9; addition of state notes; inclusion of calculations for average response factors, linear calibration, and correlation coefficient; addition of corrective actions in section 11.3 through 11.8; Clarifications in sections 12.0 & 13.0. Ohio VAP approved 2/23/09.
14	3/23/12	Technical and Quality Review and update. Added sections 1.3.1, 2.14 through 2.28, 10.13, and state/client notes in sections 1.0, 8.3.2, 8.5.2, and 11.10; Revised Attachments II and III and sections 1.2, 2.4, 7.1, 7.5, 7.8, 8.3, 9.1 through 9.8, 10.3 through 10.12, 11.1 through 11.9, 12.1, 14.7, 14.9, and 14.10; Incorporated previous minor revisions.
15	6/10/13	Technical and Quality Review and update. Added sections 4.5, 10.14 and 11.10, ; Deleted WY note in sections 8.0 and MN note in section 11.11.4, Revised Attachment III and sections 1.2, 6.1, 7.1, 7.4.1, 7.8, 8.3, 8.4 and 14.4.
16	10/24/14	Technical and Quality Review and update. Deleted state note in sections 2.1, 4.2.2.2, 8.0, 8.3.2, 10.5, 11.5 and 11.8; Deleted sections 7.3.3, 7.4.3 and 8.4; Revised sections 1.1, 2.4, 6.1, 7.5, 10.4 and 11.4.
17	8/5/2015	Technical and Quality Review and update. Revised Sections 8.4.4.1, 12.2, and 13.1. Added Section 13.4. Added State Note in Sections 4.2.1 and 8.4.4.1.
18	9/2/2015	Header and signature block formatting update. Added Attachment IV.
19	8/31/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.0, 1.3.1, 2.4, 4.2.2.2.2, 4.5, 6.1, 6.3.1, 6.4.3, 7.3.1, 7.3.2, 7.3.4, 7.4.1, 7.7, 8.0, 8.2, 8.3.2, 8.3.3, 8.3.4, 8.3.5, 8.3.6, 8.4, 8.4.1, 8.4.2, 8.4.3, 8.4.1, 8.5.1, 8.5.2, 9.1, 9.210.3, 10.4, 10.5, 10.6, 10.7, 10.8, 10.13, 10.14, 11.2.8, 11.4, 11.5, 11.6, 11.7, 11.8, and Attachment 2 Section 4.5. Deleted Sections 2.5 through 2.28, 6.4.2, 7.1, 9.3 through 9.8, and 11.11.1. Added Sections 6.3.3 and 7.2.4.
20	2/72017	Technical and quality review and update. Revised Sections 1.0, 1.3, 4.4.2, 8.0, 8.3, 8.3.5, 8.4, 8.4.1, 8.4.3, 10.5, and 14.1 through 14.16. Deleted Section 8.3.1. Added Sections 4.2.1.1, 8.5.3, and 10.4.
21	11/28/2017	Update as corrective action for 2017 A2LA audit. Changed ESC logo. Revised Section 3.1 and Attachment III Table 5.

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STANDARD OPERATING PROCEDURE
TITLE: BTEX GRO
ISSUER: Pace National – Mt. Juliet, Tennessee

Version	Date	Description of Revisions
22	3/2/2018	Update as corrective action for 2017 A2LA audit. Technical and Quality review and update. Revised sections 2.3, 3.3, 4.2.1, 4.2.1.1, 6.1, 6.3.2, 6.4.1, 7.2.1, 7.7, 7.8, 8.4.1, 8.4.2, 8.4.3, 8.4.5, 10.5, 14.1 through 14.10, and 14.12 through 14.16. Deleted state note in section 4.2.1. Added section 10.6.1.

Superseded Versions (Pace National):

Date	Description of Revisions
6/6/2019	Technical and quality review and update. Deleted header, footer and signature block. Revised sections 1.0, 1.1, 1.3, 1.3.1, 2.1, 3.1, 4.2.1, 4.2.2, 4.4.2, 4.4.15, 4.5, 6.1, 7.1, 7.2.2, 7.2.3, 7.3.1, 7.3.2, 7.4.1, 7.5, 7.8, 7.10, 8.1, 8.2, 8.3.1, 8.4, 8.4.1, 8.4.5, 8.5.1, 8.5.2, 8.5.3, 10.1, 10.2, 10.3, 10.5, 10.8, 10.10, 10.12, 10.14, 11.2.7, 11.2.8, 11.7, 12.1 and 12.2. Deleted sections 4.2.2.1, 4.2.2.2, 4.3, 8.3.2, 8.3.3, 8.3.4, 8.3.5, 11.2.2, 11.11, 11.11.1, 11.11.2, 11.11.3 and renumbered as necessary. Added sections 4.2.2.1, 4.2.2.2, 4.2.2.3, 4.2.2.4, 11.11 and renumbered as necessary. Revised Attachments I and II.
6/13/2020	Technical and quality review and update. Added header and footer.

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Attachment II: Routine Reporting Limits*

Compound	RL SOIL (mg/Kg) 1g sample size	RL Water (mg/L)
Benzene	0.0025	0.0005
Toluene	0.025	0.005
Ethylbenzene	0.0025	0.0005
M & P Xylenes	0.0050	0.0010
O Xylenes	0.0025	0.0005
MTBE	0.0250	0.005/ 0.001
GRO	0.5	0.10
Compound	**RL methanol (mg/Kg) extract by 5035A**	**RL Sodium bisulfate (mg/Kg)**
Benzene	0.025 (AK101 0.020)	0.0005
Toluene	0.25	0.005/ 0.001
Ethylbenzene	0.025	0.0005
M & P Xylenes	0.050	0.0010
O Xylenes	0.025	0.0005
MTBE	0.250	0.005/ 0.001
GRO	5.0	0.10

**See section 13.3.*

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Attachment III: DOD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701BA Version B.01.00 or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty PID	Clean PID.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.

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Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks, matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; or replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.

4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

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Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e., 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.7 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst-case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion

abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.

- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.

- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.20 The method blank shall be considered to be contaminated if:

- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
- If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

Table 3. LCS Control Limits – Method 8015 (MOD) Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	1263	100.7	11.1	67	134
303-04	Diesel Range Organics (DRO)	2184	85.2	15.7	38	132
307-27	Gasoline Range Organics (GRO)	1134	100.3	7.2	79	122
307-51	Motor Oil	658	72.2	11.2	39	106
84-15-1	o-Terphenyl	314	87.4	14.1	45	130

Table 4. LCS Control Limits – Method 8015 (MOD) Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	756	101	10.8	69	133
303-04	Diesel Range Organics (DRO)	1757	83.7	16	36	132
307-27	Gasoline Range Organics (GRO)	971	99.9	7.3	78	122
307-51	Motor Oil	573	76.9	12.1	41	113
84-15-1	o-Terphenyl	299	90.5	11.4	56	125
630-02-4	Octacosane	130	101.1	13.8	60	142

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 20%; **Option 2:** linear least squares regression for each analyte: $r^2 \geq 0.99$; **Option 3:** non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV; Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA
Method Blank (MB)	One per preparatory batch.	No analytes detected >1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use Table 3 and 4 limits or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of Positive Results (second column)	All results greater than the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

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QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality Program	20 May 2021, 01:02:51 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Supervisor	20 May 2021, 10:28:18 AM	Approved
[redacted]	Manager - EHS	20 May 2021, 10:50:06 AM	Approved
[redacted]	Manager - Operations	20 May 2021, 11:05:38 AM	Approved

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STANDARD OPERATING PROCEDURE
TITLE: Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)
ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of volatile organic compounds by EPA methods 8260B, 8260C, 8260D, 624.1, Standard Method 6200B, GRO, or similar volatile GC/MS analyses. This procedure is applicable to nearly all kinds of samples, regardless of water content, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The compounds that can be determined using this SOP are listed in Appendix C, which contains a list of the typical primary and secondary ions used in determining these compounds.

1.1 Target Analyte List and Limits of Quantitation (LOQ).

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Specific instructions for employees of the Pace Analytical National Center for Testing & Innovation (Pace National) Sacramento, California laboratory are included in this document as “SAC Notes”.

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0099.

CLIENT NOTE: For clients, whose environment laboratory quality program is administered by Environmental Standards Inc. (ESI), see controlled document QUA-30 VOA. \\FAP\NovDiskH\QAQC\Controlled Docs

2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOCs) are determined from a 5mL sample withdrawn from a sealed 40mL vial. For water samples analyzed for low levels of analytes using Method 5030 (ENV-SOP-MTJL-0131), the entire vial is placed into the instrument autosampler. The autosampler purges 5mL of sample and adds 1µL of surrogate standards and internal standards. An inert gas is bubbled through a sparger needle inserted into the sample. The purged volatile components then travel via a transfer line to a sorbent trap. When purging is complete, the trap is rapidly heated. The trap is backflushed with a helium carrier gas, to transport the desorbed sample components into a gas chromatographic (GC) column. The GC column separates and carries the components

to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.

METHOD NOTE: For Method 624.1, different sample sizes in the range of 5–25mL are allowed in order to meet differing sensitivity requirements. Calibration and QC samples must have the same volume as field samples.

2.2 For other samples, Method 5035 (ENV-SOP-MTJL-0129), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.

3.0 INTERFERENCES

3.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components must be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.

3.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.

3.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations, one or more instrument blanks may be analyzed to check for cross contamination. Alternatively, when analysis of a blank is not possible prior to the next sample, such as when an unattended autosampler is employed, the analyst should review the results for at least the next sample after the high-concentration sample. If analytes in the high-concentration sample are not present in the subsequent field sample, then the lack of carryover has been demonstrated.

3.4 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.

3.5 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C.

3.6 In extreme situations, the whole purge and trap device may require dismantling and cleaning.

3.7 Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by using a portable PID or equivalent.

3.8 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Appendix C and are recommended from the published 8260 methods.

3.9 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric

sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst must be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.10 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. A storage blank must be analyzed every two weeks to check for cross contamination of samples while samples are stored in the volatiles laboratory walk-in cooler. The storage blanks are purchased from a vendor and must matrix match the contents of the cold room or refrigerator and is placed in the cooler for a period of two weeks. Every two weeks, the applicable storage blanks are analyzed to verify that no contamination of client samples has taken place due to contamination in the storage unit.

3.11 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately 50 times higher due to poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

3.12 Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If a large amount of effervescent gas is generated, the sample may lose a significant amount of volatile analytes. If a sample effervesces, an unpreserved sample will be collected to eliminate volatiles loss whenever possible. The holding time for unpreserved VOC samples is seven days, rather than 14 days.

3.13 An analyst may re-analyze any sample if instrumentation or human error is suspected. This includes all QC samples, which can only be re-analyzed twice. If failure continues, instrument maintenance must be performed and/or the instrument must be re-calibrated.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

4.1 Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.

5.0 HEALTH AND SAFETY

5.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

5.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data

sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

5.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

5.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

5.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

5.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

5.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with laboratory SOPs ENV-SOP-MTJL-0067, *Wastewater Sampling*, ENV-SOP-MTJL-0068, *Groundwater Sampling,* and ENV-SOP-MTJL-0313, *Field Sampling*. Refer to these SOPs for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-MTJL-0064, *Sample Shipping*. For this test method, immediately after sample collection, samples should be checked for pH and Chlorine and field treated. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)
ISSUER: Pace National – Mt. Juliet, Tennessee

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Water	40mL vials (in duplicate)	Two 40 mL vials headspace free,3 vials preferred	pH<2 HCl; ≤6°C; Ascorbic acid or Na2S2O3 if Cl present	14 Days (Method 624: 7 days for aromatics if unpreserved)
Soil	4oz. w/zero headspace; 3 5g samples; 5g or 25g collected in an Encore or equivalent sampling device	5 g	pH<2 NaHSo4; Methanol	14 Days

[1]*Minimum amount needed for each discrete analysis.*

Field / Matrix QC

Trip Blank	Equipment Blank	MS/MSD	Field Duplicate
1/cooler	Per Sampling & Analysis Plan	1 pair at 5% Frequency	Per Sampling & Analysis Plan

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-MTJL-0060, *Sample Receiving*. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

6.1 Volatile analysis for water and methanol preserved soil samples must be completed within 14 days from the time of sample collection. Water samples that are not chemically preserved must be analyzed within seven (7) days. It is also a Pace National requirement that water samples with 2-Chloroethyl vinyl ether (2-CEVE), as a compound of interest, be collected unpreserved and analyzed within 7 days of collection. It has been shown that the acid preservative reacts with the 2-CEVE, which could result in false negative reporting of 2-CEVE in samples. Unpreserved soil samples must be analyzed within 48 hours from the time of collection, added to preservative or otherwise frozen at ≤-7!C. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (ENV-SOP-MTJL-0129).

STATE NOTE: The State of South Carolina requires that all soil samples must be collected and analyzed using Method 5035. Samples must be preserved within 48 hours from the time of collection, if collected in Encore™ type sampling devices. The holding time for soil samples preserved with methanol or sodium bisulfate is 14 days from the time of collection. Non-Preserved South Carolina VOCs require 7-day holding time.

6.2 Aqueous samples must be collected in at least duplicate in 40mL vials with minimal or no headspace to minimize the loss of highly volatile analytes. Sample kits can be configured to request additional vials per client request. Typically, the pH is adjusted to <2 with HCl, but the preservation requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. An example of this is some samples from Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH>12.

6.3 Soil samples may be collected by one of the following: 1) A 4oz. soil jar filled with soil with zero headspace, 2) Two 5g samples preserved in the field with 5mL $NaHS0_4$ to a pH<2 and one 5g sample preserved in the field with methanol (for high level analysis or data generated on Agilent™ 5977A or 5977B instruments [with an extractor ion or high efficiency source] may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments) or 3) A 5g or 25g sample collected in an Encore or equivalent type sampling device and frozen in the laboratory within 48 hours from the time of collection.

For all soil samples, a 4oz. soil jar should also be collected to determine percent solids and for screening purposes. All samples and extracts must be shipped and stored at <6°C.

STATE NOTE: Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH>12.

STATE NOTE: For Alaska samples, when using a water miscible solvent (e.g., methanol) to extract soil volatile organic compounds (VOC), the adjustment of solvent volume for soil moisture content must be performed. Significant soil moisture can add to a pronounced dilution when performing methanol extractions. The potential under reporting of volatile concentrations is more pronounced as the percent moisture content increases. See section 10.2.3 for the calculation.

6.4 Method 624.1 Considerations

6.4.1 If acrolein is to be determined, analyze the sample within three (3) days. To extend the holding time to 14 days, acidify a separate sample to pH 4–5 with HCl.

6.4.2 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. To extend the holding time for aromatic compounds to 14 days, acidify the sample to approximately pH 2.

6.4.3 If halocarbons are to be determined, use an acidified sample.

6.4.4 Ethers are prone to hydrolysis at pH 2 when a heated purge is used. Aqueous samples should not be acid preserved if ethers are of interest or if the alcohols, they would form upon hydrolysis are of interest and the ethers are anticipated to present.

6.4.5 2-Chloroethyl vinyl ether is subject to hydrolysis at low pH; therefore, determine 2-Chloroethyl vinyl ether from the un-acidified sample.

7.0 EQUIPMENT AND SUPPLIES

The operation, cleaning, and scheduled maintenance procedures, as prescribed by the equipment manufacturer, are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each analytical system.

7.1 Equipment

7.1.1 Instrumentation: All instrumentation meets or exceeds EPA method requirements. There are a total of 46 instruments; GCs include models 5890, 6890, 7890a, 7890b, 8890b, Intuvo or equivalent and MSs include 5973, 5975, 5977a, 5977b, HES (High efficiency source) or equivalent. Specific information for each instrument is included in the associated maintenance logbook.

Sample introduction system: Archon Autosampler, EST Centurion, OI 4760, Encon P & T, EST Evolution, OI 4660 or equivalent

7.1.2 Analytical Balance, capable of weighing to 0.01g, or equivalent

7.2 Supplies

7.2.1 Glassware: volumetric glassware equipped with penny head ground glass stopper. The volumetric flasks are cleaned by rinsing with methanol and laboratory reagent water. The volumetric flasks can be dried in a low temperature oven at less than 120°C and are never cleaned with a brush or strong alkali solution.

7.2.2 The carrier gas used for volatiles analysis is Helium-5.0 grade.

7.2.3 Syringes used for preparing the calibration curve and preparing samples and sample dilutions are Hamilton brand (or equivalent). Syringe sizes used are 0.50µL, 10µL, 25µL, 50µL, 100µL, 250µL, 500µL, 1mL, and 5mL.

7.2.4 Glass Sample (VOA) and Standard Vials

7.2.4.1 40mL VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.

7.2.4.2 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards)

7.2.4.3 2mL amber vial with Teflon™/silicone/Teflon™ septa (used to store unused standards)

7.2.5 Miscellaneous

7.2.5.1 Stainless Steel Spatula or wooden tongue depressor

7.2.5.2 Teflon™-coated stir bars, 8mm x 16mm

7.2.5.3 Glass beads –VWR™ #EM1.04015.0500, or equivalent

7.2.6 Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100!C, or equivalent

8.0 REAGENTS AND STANDARDS

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital

archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted. Working standards for most compounds should be replaced after four weeks unless the integrity of the standard is suspected to of being compromised prior to that time. Working standards for gases should be replaced after one week unless the acceptability of the standard can be documented.

8.1 Reagents

8.1.1 Nanopure water or equivalent: Nanopure water is used in all blanks to ensure it contains less than the method detection limit (MDL) of all compounds of interest. If volatile compounds are detected in the blank above MDL all samples associated with this blank must be flagged (see Section 11.3.1.1).

NOTE: For all DoD samples the laboratory water is used in all blanks to assure that it contains less than 1/2 LOQ of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds. If volatile compounds are detected in the blank above 1/2 LOQ, then those samples must be flagged.

8.1.2 Methanol, CH_3OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

8.1.3 Sodium Bisulfate, Na2S2O3 - QEC Level 3 Certified, or equivalent

8.2 Standards

8.2.1 STOCK SOLUTIONS – Primary and Secondary Sources

- # Stock calibration solutions must be purchased as certified solutions.
- # Certificates must be kept on file.
- # All Stock standards must be stored ≤ 6°C or as recommended by the standard manufacturer.
- # All non-gas stock standards must be replaced after six months, or sooner, if check standards indicate a problem.
- # Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to a second source ICV, or Secondary Source Verification Standard (SSCV).
- # Gas intermediate/secondary standards must be replaced weekly, or sooner, if comparison to check standards indicates a problem.
- # Non-gas intermediate/secondary standards must be replaced after six months, or sooner, if comparison to check standards indicates a problem.

8.2.1.1 Primary Source - Primary source standards are used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the continuing calibration verification (CCV) standard, LCS and matrix spikes. The CCV is analyzed to verify the initial calibration and is prepared using the primary source standard used to produce the calibration curve. See Section 9.2.3.2.4 through 9.2.3.2.8 for the instrument preparation of the calibration standards. When primary standards are consumed, new standards must meet the same QC criteria as the consumed standards. Stock standards must be stored below -10°C and have a six-month holding time once opened. The expiration date of the diluted standards must not exceed the expiration

date of the stock standards from which they are prepared. Once diluted, the standard must be replaced weekly. The standard list of target LCS compounds are those compounds listed in Appendix I. The LCS must be prepared in the appropriate matrix (organic-free reagent water or purified solid) depending upon the matrix within the analytical batch; and contain all of the method target analytes. A subset of the method target analytes could be used based on the project specific requirements.

STATE NOTE: South Carolina DHEC and the USACE require that all target analytes are present and evaluated in the LCS

Calibration Mix

The calibration mix is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Phenomenex	Revised 8260 Calibration Mix	AL0-130502	1	100
Phenomenex	Custom Ketones Mix	AL0-130115	1	500
Phenomenex	Acrolein	AL0-130159	1	500
Phenomenex	Custom 8260 Additions Mix	AL0-180004	1.25	100
Phenomenex	Vinyl Acetate	AL0-130117	1	500
Phenomenex	2-Chloroethyl vinyl ether	AL0-130116	1	500
Phenomenex	Ethanol Standard	AL0-130551	0.5	5000
AccuStandard	Vinyl Bromide	S-2688A	1.25	100
Agilent	Custom Standard (1,3,5-TCB)	CUS-0002027	1.25	100

8.2.1.1.1 The calibration standard mix is prepared by making a 1:1 ratio of the gas standard (Agilent, ULTRAgold™ Custom Standard CUS-27747 or equivalent at 100µg/mL) and the calibration mix standard.

8.2.1.1.1.1 For soil autosamplers (5mL), a dilution of 10x is required for the ICV mix. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is below ≤6° C.

AP9/Oxygenates Calibration Mix

The AP9/Oxygenate ICV solution is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Ethyl Acetate	681	0.125	50
NSI	Custom AP9 Standard	Q-6603-O	3.125	50
Absolute	Isopropanol	70941	0.625	25
NSI	Tert-Butyl formate	321	0.625	50

8.2.1.2 Secondary Source - Secondary source standards must be used to prepare the secondary source verification standard (SSCV) or initial calibration verification (ICV). These standards are purchased from a different vendor or the primary vendor can supply different lot numbers, if a separate vendor is not available. The standard is at a concentration near the mid-level calibration standard. Stock standards must be stored at or below ≤6°C and have a six-month holding time once opened, except the ICV

gases which have 1 week holding time. Once diluted, the standard must be replaced weekly.

SSCV

Prepare the SSCV mix in methanol in a 10mL volumetric flask as follows. A separate source or separate lot number is used for standard verification. The standard list of target LCS compounds are those compounds listed in Appendix I.

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	8260 Custom Mix 2	Q-6354-O	0.4	500
Restek	Acrolein	30645	1	500
SPEX CertiPrep	AZ fuel additive 4 comps.	VO-ESCTN-9	0.8	100
Agilent	Custom Standard	CUS-30388	0.4	Varies
Agilent	Custom Standard (ethanol)	CUS-30649	0.2	5000
Restek	1,3,5-Trichlorobenzene	31081	1	100
SPEX CertiPrep	Vinyl Bromide	S-4049	1	100

8.2.1.2.1 The working second source (SSCV) or Initial Calibration Verification (ICV) is prepared by making a 1:1 ratio of the gas standard (Phenova, VOA Gas Calibration Mix AL0-130108 or equivalent at 200µg/mL) and the second source standard.

8.2.1.2.2 For soil autosamplers (5mL), a dilution of 10x is required. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is at or below -10° C.

AP9/Oxygenates SSCV

The AP9/Oxygenates SSCV is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Custom AP9 Standard	Q-6603-O	1.25	50
NSI	Ethyl Acetate	681	.05	50
Absolute	Isopropanol	70941	0.25	25
NSI	Tert-Butyl formate	321	0.25	50

8.3 Surrogate standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below ≤6°C and have a six-month holding time, once opened. Surrogate spiking solutions are purchased from Phenova, Part# AL0-130491, or equivalent, at 20,000ug/mL, which contains both internal standards and surrogate compounds. This solution is then diluted by 100X to obtain a 200ug/mL working solution.

8.3.1 The following are Pace National designated volatiles' analysis surrogates:

Toluene-d8
4-Bromofluorobenzene

1,2-Dichloroethane-d4

8.4 Internal standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below ≤6° C and have a six-month holding time, once opened. [Internal standard and surrogate standard – Phenova, Part# AL0-130491, or equivalent]

8.4.1 The following are Pace National designated volatiles' analysis internal standards:

1,4-Dichlorobenzene-d4
Fluorobenzene
Chlorobenzene-d5

SAC NOTE: Internal Standard/Surrogate Mix – o2si 8260 Internal Surrogate Solution Catalog No.: 121722-08-20PAK. This mix is diluted 250x to produce a 50 µg/mL internal standard and surrogate mix. During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L. The internal standards and surrogates are the same as listed in 8.3.1 and 8.4.1 with the addition of Tert-butyl Alcohol-d10 as an internal standard.

8.5 4-Bromofluorobenzene (BFB) standard - The BFB in the custom internal standard mix is used to verify mass spectrometer tuning. Since internal standards and surrogates are added to all samples and standards, BFB is included as part of our initial calibration and calibration verification standards. Certificates of analysis must be kept on file. Stock standards must be stored at or below ≤6°C and have a six-month holding time, once opened.

8.6 Matrix spike (MS) standard - Stock standards must be stored at or below ≤6°C and have a six-month holding time, once opened. Once diluted, the standard must be replaced weekly.

8.6.1 The matrix spike standard is prepared from the stock standard in Section 8.2.1.

8.6.2 The spike should be at a mid-level of the calibration range. Some contracts may require a site-specific concentration.

8.6.3 Standard spiking practice requires the use of ALL TARGET ANALYTES as specified in Appendix I and must be evaluated against the current control limits presented in the LIMS.

All compounds in the spike solution must be evaluated for acceptable recovery. In the absence of established control limits, default recovery limits are 70 - 130%.

9.0 PROCEDURE

Analysis Summary**:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler, as described on Section 2. Soils require method 5035 for sample preparation, See ENV-SOP-MTJL-0129.

9.1 Equipment Preparation

Chromatographic conditions: All changes in analytical conditions are listed in the Maintenance Log.

9.1.1 Support Equipment

All support equipment used must be calibrated or verified prior to use according to SOP ENV-SOP-MTJL-0039, *General Analytical Balance Operation and Verification in the Laboratory* current revision or replacement.

Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system by screening of 5mL of sample using an HNU or equivalent portable PID. See ENV-SOP-MTJL-0102, Volatile Organic Compounds Screening using the RAE Systems Photoionization Gas Detector Model MiniRAE 3000.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions

9.1.2.1.1 Typical conditions for each instrument and column are listed below:

Inlet	off
Detector	200°C
Oven Equib. Time:	0.50 minutes
Oven Max	240°C
Init Temp	45°C hold 1.0 minute
Ramp	20°C/min to 240 hold 1.0 minute

9.1.2.1.2 Typical conditions for each autosampler are listed below:

Heating sample	1 minute at 40oC
Purge	11 minutes at 40oC
Desorb	1 minutes at 250oC
Bake	2 minutes at 260oC

9.1.2.1.3 Typical conditions for each MS detector are listed below:

Electron energy – 70 volts (nominal)
Mass range – 35 to 300 amu
Scan time – 1.2 sec/scan
Manifold vacuum – 3 x 10-6 torr

9.1.2.1.4 TUNING - Each GC/MS system must be hardware-tuned (1µL < 50ng) with BFB to meet the criteria listed below. The mass-spectrometer must meet acceptable BFB sensitivity criteria before analysis can begin. The instrument must be tuned every 12 hours for 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to each calibration.

BFB Key Ions and Ion Abundance Criteria for 8260B, 8260C, 8260D, 624.1	
Mass	**Ion Abundance Criteria**
95	50 - 200% of mass 174
96	5 - 9% of mass 95
173	< 2% of mass 174
174	50 - 200% of mass 95
175	5 - 9% of mass 174
176	95 - 105% of mass 174
177	5 - 10% of mass 176

BFB Key Ions and Ion Abundance Criteria for 6200	
Mass	**Ion Abundance Criteria**
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

EPA 624.1 NOTE: The 12-hour tune clock begins after analysis of the BFB, the LCS, and the BLANK and ends 12 hours later. BFB, the LCS, and BLANK are outside of the 12-hour tune clock. The MS and MSD are treated as samples are analyzed within the 12-hour clock.

9.2 Initial calibration

9.2.1 Calibration Design

Calibration Levels for single analytes

Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of Calibration Mix (See Section 8.2.1.1) into 5mL of reagent water. Surrogate standard is prepared by diluting the surrogate by1:10 using (NSI lab solutions, 8260 Surrogate Mix Q-4392 or equivalent at1000µg/mL).

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)
ISSUER: Pace National – Mt. Juliet, Tennessee

8260 Calibration Curve - GC/MS Soil (into 5mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.025	0.25	0	n/a
0.05	0.5 (LOD Point)	0	n/a
0.1	1	1	1
0.2	2	2	2
0.5	5	3	3
2.5	25	4	4
7.5	75	5	5
10	100	6	6
20	200	7	7

8260 Calibration Curve - GC/MS Soil (into 50mL water) for use with Agilent 5977A or 5977B Only			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.02	0.02 (LOD Point)	0	n/a
0.04	0.04	0	n/a
0.1	0.1	0	n/a
0.2	0.2	0	n/a
0.5	0.5	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6

AP9/Oxygenates Calibration Curve - GC/MS Soil (into 5mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
0.5	0.5
1	1.0
5	5
10	10
15	15
20	20

AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
5	0.5
10	1.0
50	5
100	10
150	15
200	20

Note 1: When analyzing soil samples by the low-concentration method (Section 9.4), the calibration standards must be heated to 40°C ± 1°C prior to purging.

Note 2: Injections should be performed from the lowest to the highest standards with a cleanup injected after the highest standard and followed by the secondary source standard to verify the initial calibration curve.

Water Samples - Water samples are run with a heated purge using the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of Calibration Mix (See Section 8.2.1.1) into 50mL of water. Surrogate standard is added to the curve points, (Phenova, Custom IS-SURR Mix AL0-130574 or equivalent at1000µg/mL).

8260 Calibration Curve - GC/MS Water (into 50mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.02	0.02	0	n/a
0.04	0.04	0	n/a
0.1	0.1	0	n/a
0.2	0.2	0	n/a
0.5	0.5 (LOD Point)	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6
200	200	7	7

AP9/Oxygenates Calibration Curve - GC/MS Water (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
5	0.5
10	1.0
50	5
10	10
150	15
200	20

SAC NOTE: See Controlled Document DAV-03 VOA ICAL Reference for calibration table.

9.2.2 Calibration Sequence

Curve – General Criteria

A minimum of 5-point calibration is performed using the primary standards listed in Section 8.2.1.1. Additional levels may be included to better meet project or client requirements. Regardless of the specific number, the calibration levels analyzed should correspond to a range of concentrations expected to be found in samples, without exceeding the working range of the GC/MS system.

A calibration point must be analyzed at or below the reporting limit. The concentration of the lowest calibration standard analyzed should be at least 3-5 times the MDL. The instrument response must be distinguishable from the instrument background noise. The signal to noise ratio is the magnitude of the signal strength detected by the mass spectrometer relative to the magnitude of the background noise of the instrument. Instrument conditions must be optimized before the analysis of a calibration curve to minimize background effects.

STATE NOTE: The reporting level standard must be refit after calibration is complete. This standard is required by the state of North Carolina and is used to verify the low end of the calibration curve.

STATE NOTE: When analyzing samples from Minnesota, the reporting limit must be verified with each calibration or at least monthly. Verification can be performed by re-quantitation of the low calibration standards using the newly updated calibration curve or by analyzing a separate reporting level standard following calibration curve update. This standard must recover ±40% of the expected concentration. If the criterion is not met, a higher-level standard may be re-quantitated or analyzed; however, the reporting limit must be amended to reflect the increased concentration of the standard utilized. Analytes known to be poor performers are dealt with on a case-by-case basis.

The highest standard must not exceed the linear range of the detector. The concentration of the highest standard must produce a response, which does not cause the MS detector to become saturated. The highest concentration used in the calibration curve must allow the analyte to meet the calibration requirements outlined in Sections 9.2.3.

When using Method 5035, ENV-SOP-MTJL-0129, the calibration curve must be prepared in the same solutions used to preserve the field samples.

EPA 8260C NOTE: The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to reanalyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ±30% of the standard's true concentration.

EPA 8260D NOTE: The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. A minimum quantitation check on the viability of all calibration points should be performed by re-fitting the response from each calibration standard back into the curve. The recalculated concentration of the reporting level point should be within ±50% of the standard's true concentration. All other recalculated concentration calibration points should be within ±30% of the standard's true concentration. If a failure occurs in the low point and it is equivalent to the LLOQ, the analyte should be reported as estimated near that concentration or the LLOQ should be reestablished at a higher concentration.

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

The method reference spectra must be updated from the mid-point of each calibration.

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

9.2.3.1.1 Linear Regression - Criteria

When any compound does not meet the calibration criteria for RF, the most appropriate curve fitting model is used. If linear regression is used, it must be noted on the data (preferably on the CCV RF report), next to the affected compound. It must also meet correlation coefficient (r) criteria of 0.995 or better. SM

6200 requires a linear regression correlation coefficient of >0.994.

Linear regression is achieved by plotting the instrument response versus the concentration of the standards. The resulting regression line must not be forced through the origin and the origin must not be included as a calibration point.

STATE NOTE: For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40% RSD is used for normal target analytes and 50% RSD is utilized for known poor performing compounds.

The most appropriate curve fitting model from among the following choices must be utilized (given in the order of preference): Average Response Factor

Linear – No Weighting
Linear – 1/x Weighting
Linear – $1/x^2$ Weighting
Quadratic

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

9.2.3.1.2 Calibration Corrective Action

When the RSD exceeds 15% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

If calibration criteria are not met, then one of the following options must be applied to the GC/MS initial calibration:

9.2.3.2 Relative Standard Error (RSE)

A measure of relative error must be documented for each calibration. For analytes using an Average curve fit, the %RSD is the relative error, and no further evaluation is required. For analytes using a linear or quadradic curve fit, the relative error must be evaluated for the calibration low and mid-points using one of the procedures below.

9.2.3.2.1 %Relative Error (%RE)

If using this method of determining relative error, the %RE of the calibration mid-point must be between 70-130%. For the calibration low point, %RE must be between 50-150%. The %RE

is evaluated busing on the response/resultant concentration in the applicable initial calibration standards.

9.2.3.2.2 %Relative Standard Error (%RSE)

If using this method of determining relative error, the acceptance limits for %RSE are numerically identical to the %RSD criteria. The %RSE is evaluated by creating a copy of the applicable calibration standard files and then quantitating the copied files against the new calibration.

Note: Calibration standard files must be copied and re-named for use in this evaluation. DO NOT over-write the initial calibration files.

Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.

9.2.3.2.3 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Re-calculate the RSD without the low standard to determine if the RSD meets the QC limit. If the lowest standard is dropped, the reporting limit could require a change. Check with the supervisor to determine if a point can be removed and not affect reporting limits requirements.

Compounds that are very soluble in water generally are poor purgers. The ketones, vinyl acetate, acrolein, and acrylonitrilie fall into this category.

9.2.3.2.4 EPA 8260B: Response Factors (RF's) & Calibration Check Compounds (CCC's) - Soil/Water

Using the RFs for the initial calibration curve from Section 8.2.2, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the percent RSD as in Section 10.2.5. Linearity can be assumed if the RSD criteria is met, thus allowing quantitation calculations to be performed using RF.

CCC Criteria - The %RSD for each individual CCC must be less than 30%. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

Target Analytes and other Non-CCC's - The RSD must meet the following criteria

<15% RSD for all 8260B Target Analytes
<20% RSD for all 8260C/D Target Analytes
<35% RSD for all 624.1 Target Analytes
<20% RSD for all KSGRO Samples
<15% RSD for n-Hexane
<20% RSD for 6200 Analytes
<15% appendix 9 Analytes
<10% RSD for all 601/602 Target Analytes

Compounds not meeting the RSD requirement may be considered for linear regression as stated in 8.2.7.3

9.2.3.2.5 EPA 8260C: Response Factors (RF's)

9.2.3.2.5.1 Calibration Curve Criteria - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes as noted in Appendix G, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.

9.2.3.2.6 When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%.

9.2.3.2.7 EPA 8260D: Response Factors (RF's)

9.2.3.2.7.1 Calibration Curve Criteria – Calculate the mean RF and the relative standard deviation (RSD) of the RFs for each target analyte. The RSD should be <20% for each target analyte. Appendix H contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if

calibration standards are prepared correctly. It is neither expected nor required that all analytes meet these minimum RFs in the guidance table, however target analytes must not have an RF <0.01. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.

9.2.3.2.7.2 When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses as the inspection can be a useful diagnostic tool.

NOTE: To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%

9.2.3.2.8 EPA 624.1

Calculate the mean (average) and relative standard deviation (RSD) of the response factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used the regression must be weighted inversely proportional to concentration. The coefficient of determination (R2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

NOTE: Using capillary columns and current instrumentation, it is quite likely that a laboratory can calibrate the target analytes in this method and achieve a linearity metric (either RSD or RSE) well below 35%. Therefore, laboratories are permitted to use more stringent acceptance criteria for calibration than described here (e.g., to harmonize their application of this method with those from other sources).

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

9.2.3.3 Initial Calibration Verification

Calibration Verification for EPA Methods 8260B, 624.1 and SM 6200B:

9.2.3.3.1 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

CCC and SPCC compounds	± 30%
Other compounds (non-poor performers)	± 40%
Poor Performers (8.3.2	in-house LCS limits

9.2.4 Continuing Calibration Verification

9.2.4.1 Internal Standards and Surrogates – Soil/Water

The autosampler adds 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution is equivalent of 16µg/L of each surrogate/internal standard. Internal standard and surrogate standard are contained within the same spiking mix. Internal Standards are listed Section 8.4.1 and Surrogates are listed in Section 8.3.1.

SAC NOTE: During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L

Tabulation of the Internal Standards

Tabulate the area response of the characteristic ions (see Appendix C) against each compound's concentration and each internal standard concentration. Then calculate the response factor (RF) for the quantifying ion of each compound relative to the appropriate internal standard according to the calculation provided in Section 9.1. The internal standards used should permit most of the compounds of interest in a chromatogram to have retention times of 0.80 to 1.20, relative to one of the internal standards. The average RF must be calculated and recorded for each compound.

9.2.4.2 System Performance Check Compounds (SPCCs) – Soil/Water

A system performance check must be made before the calibration curve can be used. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichlorethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

These compounds are typically used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

Compound	Effect on stability
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachlorethane and 1,1-Dichloroethane	Contaminated transfer lines degrade these compounds in purge-and-trap systems. Active sites in trapping materials also can cause problems.

Adjust the purge gas (Helium or Argon) flow rate to 25-40mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

9.2.4.3 CCC's

The curve must be verified daily by a calibration standard known as the Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours. This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for CCC analytes (Listed in Section 8.2.7.1) and n-Hexane when requested as a target analyte.

Compounds on average response factor use % difference,

$$\% \text{ Difference} = (RF_v - Rf_{ave}) / Rf_{ave} \times 100\%$$

Compounds on regression fit model use percent drift,

% Drift = (Calculated conc - Theoretic conc) / Theoretic conc x 100%

Criteria for both is $\leq$20%.

9.2.4.4 SPCC's

The SPCC's must have a minimum response factor as stated in Section 8.2.6. If these criteria are exceeded, then corrective action is required.

9.2.4.5 All Target Analytes and Non-CCC's

When analyzing 8260B and 624.1 concurrently, calibration verifications are evaluated using both the 8260B criteria (sections. 8.3.1.2 through 8.3.1.3 and the 624.1 criteria (Appendix F). For analytes not on the 624.1 list, all target analytes (except for the poor performers (9.2.4.7) must meet a maximum of 40% drift from the calibration curve. The analyst evaluates all analytes carefully and the experience of the analyst weighs heavily when determining the usability of the data.

Poor performers are allowed a maximum of 50% drift from the calibration curve. See section 9.2.4.7 for a listing of poor performing analytes.

STATE NOTE: For South Carolina 624.1 samples, the target analytes reported must agree with the compound list found in the EPA 624.1 published method.

Calibration Verification for EPA Method 8260C/D:

9.2.4.6 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

All compounds	± 30%
Poor Performers (9.2.4.7)	in-house LCS limits

9.2.4.7 Target Analytes

The curve must be verified initially by a calibration standard known as the Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours). This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for all target analytes and n-Hexane when requested as a target analyte.

If the percent difference (average RSD) or percent drift (linear regression models) for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, a reporting level verification is analyzed. Reference QUA-12 *CCV Criteria for 8260C and 8270D* for assessing reporting level verification.

All compounds must have a minimum response factor. If these criteria are exceeded, then corrective action is required. The minimum response factors are generated in house based off historical data.

Poor Performers:

The poor performers are as follows:

Propene	2-Chloroethylvinyl Ether
Dichlorodifluoromethane	Acrolein
Carbon Disulfide	Vinyl acetate
Bromomethane	trans-1, 4-dichloro-2-butene

Chloroethane	Alcohols (Ethanol, TBA, TAA, ETBA, Butanol)
1,3-Butadiene	Iodomethane
1,2-Dibromo-3-chloropropane	Naphthalene
1- Methylnaphthalene	2-Butanone
2- Methylnaphthalene	2-Hexanone
Acetone	4-Methyl-2-pentanone
Pentachloroethane	Cyclohexanone
Tert-butyl Formate	Methyl cyclohexane

9.2.4.8 Laboratory Control Standard (LCS): A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Also note the LCS for water sample matrices is typically prepared in organic-free reagent water similar to the continuing calibration verification standard. In these situations, a single analysis can be used for both the LCS and continuing calibration verification.

QC Limits are found in the LIMS for LCS and MS/MSD.
If the stated criteria are exceeded, then corrective action is required.
See Section 12.9.1, on marginal exceedances.

STATE NOTE: All **South Carolina** DHEC compliance testing, the LCS responses must be within 70 – 130% for Method 8260 and within the limits given in Appendix F for Method 624.1. High failures are acceptable for 8260 or 624.1, as long as the sample results are below detection level. Qualifiers cannot be used therefore low failures require a batch re-analysis. See Section 11 for QC evaluation. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

STATE NOTE: For EPA Region IV only (AL, FL, GA, KY, MS, NC, SC, TN), when running Acrolein and Acrylonitrile by V624.1, the QC criteria from EPA Method 603 should be used for control demonstration. The LCS criteria from EPA Method 603 include Acrolein recovery within 88-118% and Acrylonitrile recovery within 71-135%.

9.2.4.9 Internal Standard Evaluation

When a calibration is performed at the beginning of an analytical run:

The internal standard areas must be evaluated against the mid-point of the curve. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve.

Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Addition samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily CCV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

9.3 Sample Preparation

Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system by screening of 5mL of sample using an HNU or equivalent portable PID.

9.3.1 Homogenization and SubSampling

Compositing samples prior to GC/MS analysis – Site or project-specific requirements may require compositing of samples, which is performed according to the instructions below. Compositing of samples is only performed at the request of the client.

9.3.1.1 All vials for the sample are combined in an appropriately sized volumetric flask that will allow for the least amount of headspace. (If 4 vials are to be composited then a 200mL volumetric flask will be used to combine the samples.). Practice special precautions to maintain zero headspace in the syringe.

9.3.1.2 The samples must be cooled to 4°C or less during composition to minimize the loss of volatiles. Sample vials may be placed in a tray of ice to prevent volatile loss during this process.

9.3.1.3 Invert the volumetric flask three (3) times. Pour the volume out of the volumetric flask into the original 40mL VOA vial containers. The sample is now ready to be analyzed.

NOTE: Samples are not routinely composited; however, if site-specific requirements state procedures for compositing samples, the laboratory makes every effort to comply with those requirements.

9.4 Analysis

9.4.1 Example Analytical Sequence

9.4.1.1 Gas chromatographic analysis:

9.4.1.1.1 Typical sequence order for loading the autosampler with calibration:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Calibration standard(s)	Initial volatiles calibration and 5-point for GRO (if analyzed)
Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV)	Verify initial calibration with second source.
Laboratory Control Sample	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known number(s)(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

9.4.1.1.2 Typical sequence order for loading the autosampler with calibration verification only:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Initial Calibration Verification (ICV)	Verify initial calibration.
Laboratory Control Sample	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

9.4.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

9.4.3 BFB tuning criteria and GC/MS calibration criteria must be met before analyzing samples

Load the unopened VOA vial onto the autosampler for analysis.

9.4.4 After the sample has been analyzed on the instrument, check the pH of the sample using the remaining sample in the VOA vial. Use universal pH paper and record the sample pH to the nearest whole pH unit. Samples not passing the pH requirements are flagged with a “G1” qualifier. All samples that report 2-CEVE as a target analyte and have a pH < 2 are qualified with a “G2”.

STATE NOTE: For West Virginia compliance samples, check for residual chlorine after sample analysis. Samples containing residual chlorine must be flagged as such.

Sample Dilution -- When necessary, samples can be diluted before purging. This can be performed in a clean 50mL volumetric flask. The sample is measured through the use of an appropriate microliter syringes and added to the flask which is then filled with reagent water to the meniscus.

Surrogate/Internal Standards – The autosampler adds 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution is equivalent to 16µg/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

SAC NOTE: During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L of each surrogate and internal standard.

If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Secondary ion quantitation is allowed only when there are sample interferences with the routinely quantitated primary ion. When a sample is analyzed that has saturated the detector, the samples following must be analyzed for contamination. If any sample shows contamination, they must be re-analyzed.

9.4.5 GC/MS Analysis - Water-miscible liquids

9.4.5.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 25-fold with laboratory water.

9.4.5.2 Initial and serial dilutions can be prepared by pipetting a known amount of the sample to a 50mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a clean/baked 40mL vial using a 5mL syringe.

9.4.5.3 Alternatively, prepare dilutions directly in a clean 40mL vial filled with organic-free reagent water by adding at least 0.5µL, but not more than 25mLs of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 9.2.4.1.

9.4.6 GC/MS Analysis - Sediment/soil and waste samples

These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system and require extensive cleanup and instrument downtime. The screening of samples is highly recommended. Screening data should be used in conjunction with site-specific DQOs, if known,

to determine whether to use the low-concentration method (0.005 - 1mg/Kg) or the high-concentration method (>1mg/Kg).

9.4.6.1 Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. All QC samples and standards are to be analyzed under the same conditions as the samples, using 5g of glass beads or equivalent blank matrix.

STATE NOTE: This option cannot be used for South Carolina samples. Please refer to ENV-SOP-MTJL-0129 that addresses Method 5035 for sample preparation.

9.4.6.2 Use a 5g sample if the expected concentration is <0.1mg/Kg, or a 1g sample for expected concentrations between 0.1 and 1mg/Kg.

9.4.6.3 The GC/MS system must be set up prior to the preparation of the sample to avoid loss of volatiles from standards and samples. Both the initial and daily calibration standards must be heated to 40°C purge temperature.

9.4.6.4 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. Weigh the amount determined into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

9.4.6.5 Add nanopure water to the purging vial, which contains the weighed amount of sample, and place the vial in the purge-and-trap system.

NOTE: Prior to the placement of the vial, the procedures in Sections 9.4.6.5 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

9.4.7 High-concentration method -- The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e., petroleum and coke wastes) that are soluble in methanol are diluted. An aliquot of the extract is added to organic-free reagent water containing surrogate and internal standards. This may be purged at higher temperatures than ambient temperature as long as all calibration standards, field samples, and associated QC samples are purged at the same temperature and the laboratory demonstrates acceptable method performance. All samples with an expected concentration of >1.0 mg/Kg must be analyzed by this method.

STATE NOTE: This method is not suitable for samples from South Carolina, North Carolina, or Indiana. South Carolina does not recognize the practices in sections 9.4.7.1 and 9.4.7.2 5035 must be used for all high-level soil samples, see ENV-SOP-MTJL-0129.

9.4.7.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol, weigh 10g (wet weight) of sample into a tared 40mL vial. Use a top-loading balance. Note and record the actual weight to 0.1g. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 10g (wet weight) into a 40mL vial.

9.4.7.2 Add 10mL Methanol and vortex. See ENV-SOP-MTJL-0129.

NOTE: Sections 9.4.7.1 and 9.4.7.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

9.4.7.3 The GC/MS system must be set up as in Sections 9.

9.4.7.4 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 200µL.

9.4.7.5 In a clean/baked vial filled with reagent water, inject the corresponding aliquot of methanol extract. Immediately cap and place in the autosampler. The autosampler adds 1µL of the IS/surrogate mix to all of the samples.

9.4.7.6 Proceed with the analysis as outlined in Sections 9. Analyze all blanks on the same instrument as that used for the samples.

9.4.7.7 For a matrix spike in the high-concentration of sediment/soil samples, Add a 200µL aliquot of this extract to 5mL of organic-free reagent water for purging (as per Section 9.4.7.6) in a clean/baked 40mL VOA vial and add 20µL spiking solution, 1µL internal and surrogate standard solution (IS/Surr solution added by autosampler).

9.4.7.8 Data generated from soil samples prepared in methanol on Agilent™ 5977A or 5977B instruments (with an extractor ion or high efficiency source) may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits must be visually compared to the reference spectrum for confirmation. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the criteria below are met.

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.

The RRT of the sample component is within + 0.06 RRT units of the RRT of the standard component.

The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum the corresponding abundance in a sample spectrum can range between 20% and 80%.)

Structural isomers that produce very similar mass spectra are 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.

Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound**.**

10.1.1 Tentatively Identified Compounds (TICS)

TIC's - Tentatively Identified Compounds

Periodically, clients may request additional identification of compounds that are not normally calibrated. This identification is limited to the compounds in the current mass spectral library employed by Pace National.

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 is determined by the type of analyses being conducted. At the client request, when serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) must always be reported. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within 15% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum the corresponding sample ion abundance must be between 20 and 80%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(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.

(5) Ions present in the reference spectrum but not in the sample spectrum must 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.

Computer generated library search routines must 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 does the mass spectral interpretation specialist assign a tentative identification.

10.1.1.1 Routinely, Pace National employs a minimum Q value of 85% for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 85% but above 10ppb may be reported as “Unknown”. Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (e.g., siloxanes) are also removed.

10.1.1.2 If multiple TICs, with same exact name, exist for a sample, the LIMS will only display the TIC with the highest quality match per sample.

10.1.1.3 TIC names assigned as “Unknown” may initially have the same name as another “Unknown” until parsed and displayed in LIMS where it is given a hyphen and incremental number which then becomes a unique TIC (e.g., Unknown-1).

10.1.1.4 When reporting “Total TIC” for any client sample, only concentrations per above requirements will be used to sum the Total TIC concentration.

10.1.2 Manual Integration

Manual changes to automated integration are called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument

maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration.*

10.2 Quantitative Identification

When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is accomplished using the internal standard technique, as described in Section 9. The internal standard used must be the one nearest the retention time of that of a given analyte.

Sediment/soil samples are reported on a dry weight basis, while sludge and wastes are reported on a wet weight basis. The percent dry weight of the sample (see Section 9.7) must be reported along with the data in either instance. At Pace National, the dry weight conversion calculations for sample reporting are performed by the LIMS system. [Dry weight only when requested]. The LIMS Final Client Report represents the reporting basis as either wet weight or dry weight, depending upon the calculation used.

10.2.1 Concentration of Target Analytes in Water and Water-Miscible Waste

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(D)}{(A_{is})(\text{ave.RF})(V_s)}$$

where:
A_x = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
I_s = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.
D = Dilution factor if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
ave.RF = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample extracted or purged (mL). If units of liters are used for this term, multiply the results by 1000.

10.2.2 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

10.2.2.1 High-concentration procedure

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:
A_x, I_s, A_{is}, ave.RF are the same as in water and water-miscible waste above.
Vt = Volume of total extract (µL) (use 10,000µL or a factor of this when dilutions are made).
Vi= Volume of extract added (µL) for purging.
Ws = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

10.2.2.2 Low-concentration procedure

$$\text{Concentration}(ug/L) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:
A, I_s, A_{is}, RF are the same as in water and water-miscible waste above.
V_t = Volume of total extract (µL) (use 10,000µL or a factor of this when dilutions are made).
V_i = Volume of extract added (µL) for purging.
W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

10.2.2.3 Soil Weight determination with Methanol (samples received with MeOH).

$$\text{SoilWeight} = \text{VialTotalWeight(vial, soil, MeOH)} - \text{TareVialWeight} - \text{MeOHWeight}.$$

10.2.3 In order to report results for volatiles analysis of samples prepared in methanol containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as Vt. This total solvent/water volume is calculated as follows:

$$\mu L \text{ solvent/water } V_t = \left[\frac{\text{mL of solvent} + (\%\text{ moisture} \times \text{g of sample})}{100}\right] \times 1000\ \mu L/mL$$

10.2.4 Percent Error (%Error)

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

where:
x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units
x_i = True amount of analyte at calibration level *i*, in mass or concentration units

10.2.5 Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 624.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^{n}\left[\frac{x'_i - x_i}{x_i}\right]^2}{(n-p)}$$

where:
x'i = Calculated concentration at level i
xi = Actual concentration of the calibration level i
n = number of calibration points

p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

10.3 Calculations

See the Laboratory Quality Assurance Manual for equations for common calculations.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Sample Duplicate	Not required for this method
Trip Blank	As requested by client
Surrogate	All field and QC samples
Internal Standard	All field and QC samples

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Tune	Prior to ICAL and every 12-hour period of sample analysis
Initial Calibration	At instrument setup and prior to sample analysis
Initial Calibration Verification	After each ICAL prior to sample analysis
Initial Calibration Blank	After each ICAL prior to sample analysis and as needed
Continuing Calibration Verification	Daily before analysis, after every 12-hour period of sample analysis and at the end of the analytical sequence
Continuing Calibration Blank	After each CCV and as needed to verify the system is contamination free
RT Window	Once per ICAL and at the beginning of the analytical sequence
Relative Retention Time	With each sample
Breakdown Check	Not required for this method

11.3 Method Performance

11.3.1. Method Validation

11.3.1.1 Detection Limits**11.3.1.2** Method 624.1 Requirements

11.3.1.2.1 Establish MDLs for the analytes of interest using the MDL procedure at 40 CFR part 136, appendix B. The laboratory's MDLs must be equal to or lower than those listed in Table 1 for those analytes which list MDL values, or lower than one-third the regulatory compliance limit, whichever is greater. For MDLs not listed in Table 1, the laboratory must determine the MDLs using the MDL procedure at 40 CFR part 136, appendix B under the same conditions used to determine the MDLs for the analytes listed in Table 1. All procedures used in the analysis must be included in the DOC.

624.1 Purgeable Analytes	MDL (ug/L)	Limit for s (%)	Range for $\bar{X}$ (%)
Acrolein		30	50-150
Acrylonitrile		30	50-150
Benzene	4.4	33	75-125
Bromodichloromethane	2.2		
Bromoform	4.7		
Bromomethane		90	D-242
Carbon tetrachloride	2.8	26	65-125
Chlorobenzene	6.0	29	82-137
Chloroethane		47	42-202
2-Chloroethylvinyl ether		130	D-252
Chloroform	1.6	32	68-121
Chloromethane		472	D-230
Dibromochloromethane	3.1	30	69-133
1,2-Dichlorobenzene		31	59-174
1,3-Dichlorobenzene		24	75-144
1,4-Dichlorobenzene		31	59-174
1,1-Dichloroethane	4.7	24	71-143
1,2-Dichloroethane	2.8	29	72-137
1,1-Dichloroethene	2.8	40	19-212
trans-1,2-Dichloroethene	1.6	27	68-143
1,2-Dichloropropane	6.0	69	19-181
cis-1,3-Dichloropropene	5.0	79	5-195
trans-1,3-Dichloropropene		52	38-162
Ethylbenzene	7.2	34	75-134
Methylene chloride	2.8	192	D-205
1,1,2,2-Tetrachloroethane	6.9	36	68-136
Tetrachloroethene	4.1	23	65-133
Toluene	6.0	22	75-134
1,1,1-Trichloroethane	3.8	21	69-151
1,1,2-Trichloroethane	5.0	27	75-136
Trichloroethene	1.9	29	75-138
Trichlorofluoromethane		50	45-158
Vinyl chloride		100	D-218

11.3.1.3 Prepare and analyze four LCSs by adding an appropriate volume of the second source standard (Section 7.5.2.1) to each of four aliquots of reagent water.

11.3.1.4 Calculate the average percent recovery ($\overline{X}$) and the standard deviation of the percent recovery (s) for each analyte using the four results.

11.3.1.5 For each analyte, compare s and $\overline{X}$ with the corresponding acceptance criteria for precision and recovery in Section 10.1.1.1. For analytes not listed, DOC QC acceptance criteria must be developed by the laboratory. Alternatively, acceptance criteria for analytes not listed may be based on laboratory control charts. If s and $\overline{X}$ for all analytes of interest meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin. If any individual s exceeds the precision limit or any individual $\overline{X}$ falls outside the range for recovery, system performance is unacceptable for that analyte.

NOTE: The large number of analytes present, a substantial probability that one or more will fail at least one of the acceptance criteria when many or all analytes are determined simultaneously. Therefore, the analyst is permitted to conduct a ‘‘re-test’’ as described here.

When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable, and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test. To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between this pair of tests.

11.3.1.6 Batches

11.3.1.6.1 Extraction Batches:

Extraction batches are defined as sets of 1 - 20 samples. Extraction batches must include the following: 1 method blank, 1 Laboratory Control Sample (LCS)), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair (if sufficient sample is available). Exceptions are made for waste dilution samples where the minimum batch QC must include a blank, and an LCS.

Additional instructions on Batch QC including required frequency and corrective actions can be found in Section 12 while acceptance criteria are found in the LIMS.

STATE NOTE: For samples from FL, AZ, MN, and MA, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) is required per batch.

11.3.1.6.2 Analytical Batches (sequences):

Analytical batches analysis must include the following: 1 Initial Calibration Verification (ICV) and BFB tune at the beginning of run, and/or 1 Continuing Calibration Verification (CCV) and BFB tune every 12 hours.

11.3.1.7 Perform BFB tune every 12 hours for 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to calibration curve. Tuning acceptance criteria are presented in Section 9.1.2.1.4. Once the data is acquired, the following options are available for acquiring the spectra for reference to meet the BFB tuning requirements. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex &+1 scan) and average the three scans then perform background subtraction. If Autofind is not utilized, select the mass spectrum at the peak apex for evaluation, or use an average mass spectrum across the entire BFB peak. Background subtraction is conducted using a single scan prior to the elution of BFB but no more than 20 scans prior. No part of the BFB peak or any other discrete peak should be subtracted.

If a tune cannot be achieved that meets the criteria in Section 9.1.2.1.4 retune the mass spectrometer and run a new calibration or maintenance may be necessary.

11.3.1.7.1 Method 624.1 Requirements

11.3.1.7.1.1 Verify calibration after the criteria for BFB are met and prior to analysis of a blank or sample. After verification, analyze a blank to demonstrate freedom from contamination and carry-over at the MDL. Tests for BFB, the CCV, and the blank are outside of the 12-hour shift, and the 12-hour shift includes samples and matrix spikes and matrix spike duplicates. The total time for analysis of BFB, the CCV, the blank, and the 12-hour shift must not exceed 14 hours.

11.3.1.8 Run a minimum of a 5-point initial calibration curve (3-point can be used if 624/6200B are being run independently of 8260B), using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria for initial calibration are presented in Section 9.2. Calibration is verified by analyzing Second Source Calibration Verification (SSCV) standard; acceptance criteria for the SSCV are presented in Section 9.2.3.3.1.

11.3.1.8.1 Method 624.1

If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (R2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion.

As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

STATE NOTE: South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

11.3.1.9 Run a mid-point Continuing Calibration Verification (CCV) using the primary source standards every 12 hours before sample analysis and every 12 hours during an analytical sequence for 8260B, 8260C, 8260D, 624.1 and 6200B. See sections 9.2.4-9.2.4.5 for acceptance criteria.

11.3.1.10 Retention Time Evaluation

11.3.1.10.1 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 10 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.3.1.10.2 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

11.3.1.11 Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, polishing detector windows, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

11.3.1.12 METHOD BLANK - The analyst must confirm that this blank was analyzed at the required frequency of 1 per batch of 20 samples. The method blank must not exhibit any contamination of any analyte above the method detection limit for any of the method target analytes.

11.3.1.12.1 If more than one instrument blank or method blanks are analyzed, evaluate, and assess the blank and field samples under the same conditions for possible mid-level standard carryover using the subsequent blank after the mid-level standard on a per analyte basis.

11.3.1.12.2 Method 624.1 – If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at

a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample analyzed during the 12-hour shift, whichever is greater; analysis of samples must be halted, and samples affected by the blank must be re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance.

11.3.1.13 LABORATORY CONTROL SAMPLES - Assess that the LCS was prepared at the required frequency of 1 per batch of 20. If the same injection is used for the LCS and the CCV, ensure that no more than 20 samples are analyzed in conjunction. Routine LCS Control limits are presented in the LIMS.

STATE NOTE: For all samples analyzed from South Carolina, the LCS/LCSD RPD must be <20%, recoveries must be 70-130% in a soil matrix, and recoveries must be within the following limits in a water matrix:

Benzene	70 – 130%
Bromodichloromethane	70 – 130%
Bromoform	70 – 130%
Bromomethane	70 – 130%
Carbon tetrachloride	70 – 130%
Chlorobenzene	70 – 130%
Chloroethane	70 – 130%
2-Chloroethyl vinyl ether	70 – 130%
Chloroform	70 – 130%
Chloromethane	70 – 130%
Dibromochloromethane	70 – 130%
Dichlorodifluoromethane	70 – 130%
1,2-Dichlorobenzene	70 – 130%
1,3-Dichlorobenzene	70 – 130%
1,4-Dichlorobenzene	70 – 130%
1,1-Dichloroethane	70 – 130%
1,2-Dichloroethane	70 – 130%
1,1-Dichloroethene	70 – 130%
Trans-1,2-Dichloroethene	70 – 130%
1,2-Dichloropropane	70 – 130%
Cis-1,3-Dichloropropene	70 – 130%
Trans-1,3-Dichloropropene	70 – 130%
Ethyl benzene	70 – 130%
Methylene chloride	70 – 130%
Methyl tert-butyl ether	70 – 130%
1,1,2,2-Tetrachloroethane	70 – 130%
Tetrachloroethene	70 – 130%
Toluene	70 – 130%
1,1,1-Trichloroethane	70 – 130%

1,1,2-Trichloroethane	70 – 130%
Trichloroethene	71 – 130%
Trichlorofluoromethane	70 – 130%
Vinyl chloride	70 – 130%
Xylenes, total	70 – 130%

11.3.1.14 Method 624.1 Requirements

11.3.1.14.1 Compare the percent recovery (Q) for each analyte with its corresponding QC acceptance criterion in the following table. For analytes of interest not listed in the following table, use QC acceptance criteria developed for the LCS. If the recoveries for all analytes of interest fall within their respective QC acceptance criteria, analysis of blanks and field samples may proceed. If any individual Q falls outside the range, proceed according to Section 8.3.5.1.4.

624.1 Purgeable Analytes	Range for Q (%)
Acrolein	60-140
Acrylonitrile	60-140
Benzene	65-135
Bromodichloromethane	65-135
Bromoform	70-130
Bromomethane	15-185
Carbon tetrachloride	70-130
Chlorobenzene	65-135
Chloroethane	40-160
2-Chloroethylvinyl ether	D-225
Chloroform	70-135
Chloromethane	D-205
Dibromochloromethane	70-135
1,2-Dichlorobenzene	65-135
1,3-Dichlorobenzene	70-130
1,4-Dichlorobenzene	65-135
1,1-Dichloroethane	70-130
1,2-Dichloroethane	70-130
1,1-Dichloroethene	50-150
trans-1,2-Dichloroethene	70-130
1,2-Dichloropropane	35-165
cis-1,3-Dichloropropene	25-175
trans-1,3-Dichloropropene	50-150
Ethylbenzene	60-140
Methylene chloride	60-140
1,1,2,2-Tetrachloroethane	60-140
Tetrachloroethene	70-130
Toluene	70-130
1,1,1-Trichloroethane	70-130
1,1,2-Trichloroethane	70-130
Trichloroethene	65-135
Trichlorofluoromethane	50-150
Vinyl chloride	5-195

11.3.1.14.2 Repeat the test only for those analytes that failed to meet the acceptance criteria (Q). If these analytes now pass, system performance is acceptable, and analysis of blanks and samples may proceed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to repair, repeat the calibration verification/LCS test. If the acceptance criteria for Q cannot be met, re-calibrate the instrument. To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests

11.3.1.15 MATRIX SPIKE/MATRIX SPIKE DUPLICATE ASSESSMENT: Assess that matrix spike/matrix spike duplicates were analyzed at required frequency of 1 per batch of 20 if volume allows.

The analyst also verifies that the samples were spiked at the appropriate level.

The order of preference for spiking levels is as follows:

1) If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two

2) If an action level exists, spike at this level

3) If neither of the first two conditions applies, spike at a level that corresponds between the low and mid-level calibration standards.

4) All RPD results must be within the indicated control limits found in the LIMS.

Acceptance criteria are that all %Recovery and/or RPD results must be within the indicated control limits on the appropriate MS control charts. See the LIMS for LCS/LCSD & MS/MSD limits and QC acceptance.

STATE NOTE: For all water samples analyzed from South Carolina, the MS/MSD recoveries must be within the most stringent limits comparing in-house derived recovery limits to those given in Table 3 of Method 608. The following are the current water limits:

Benzene	58.6 - 133%
Bromodichloromethane	69.2 – 127%
Bromoform	66.3 – 140%
Bromomethane	16.6 – 183%
Carbon tetrachloride	70 – 139%
Chlorobenzene	70.1 – 130%
Chloroethane	33.3 – 155%
2-Chloroethyl vinyl ether	5 – 149%
Chloroform	66.1 – 133%

Chloromethane	40.7 – 139%
Dibromochloromethane	69.2 – 127%
Dichlorodifluoromethane	42.2 – 146%
1,2-Dichlorobenzene	77.4 – 127%
1,3-Dichlorobenzene	67.9 – 136%
1,4-Dichlorobenzene	74.4 – 123%
1,1-Dichloroethane	64 – 134%
1,2-Dichloroethane	60.7 – 132%
1,1-Dichloroethene	48.8 – 144%
Trans-1,2-Dichloroethene	61 – 132%
1,2-Dichloropropane	69.7 – 132%
Cis-1,3-Dichloropropene	71.1 – 129%
Trans-1,3-Dichloropropene	66.3 – 136%
Ethyl benzene	62.7 – 136%
Methylene chloride	61.5 – 125%
Methyl tert-butyl ether	61.4 – 136%
1,1,2,2-Tetrachloroethane	64.9 – 145%
Tetrachloroethene	64 – 141%
Toluene	67.8 – 124%
1,1,1-Trichloroethane	58.7 – 134%
1,1,2-Trichloroethane	74.1 – 130%
Trichloroethene	71 – 148%
Trichlorofluoromethane	39.9 – 165%
Vinyl chloride	44.3 – 143%
Xylenes, total	65.6 – 133%

11.3.1.16 Method 624.1 Requirements

11.3.1.16.1 Spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory must spike at least one sample in duplicate per extraction batch of up to 20 samples with the analytes in the table in Section 11.3.1.1.1. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.

11.3.1.16.2 If the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration, at or near the mid-point of the calibration range, or at the concentration in the LCS whichever concentration would be larger.

11.3.1.16.3 Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in the following table. A laboratory may develop and apply QC acceptance criteria more restrictive than the presented criteria if desired.

624.1 Purgeable Analytes	Range for P1, P2 (%)	Limit for RPD
Acrolein	40-160	60
Acrylonitrile	40-160	60
Benzene	37-151	61
Bromodichloromethane	35-155	56
Bromoform	45-169	42
Bromomethane	D-206	61
Carbon tetrachloride	70-140	41
Chlorobenzene	37-160	53
Chloroethane	14-230	78
2-Chloroethylvinyl ether	D-305	71
Chloroform	51-138	54
Chloromethane	D-273	60
Dibromochloromethane	53-149	50
1,2-Dichlorobenzene	18-190	57
1,3-Dichlorobenzene	59-156	43
1,4-Dichlorobenzene	18-190	57
1,1-Dichloroethane	59-155	40
1,2-Dichloroethane	49-155	49
1,1-Dichloroethene	D-234	32
trans-1,2-Dichloroethene	54-156	45
1,2-Dichloropropane	D-210	55
cis-1,3-Dichloropropene	D-227	58
trans-1,3-Dichloropropene	17-183	86
Ethylbenzene	37-162	63
Methylene chloride	D-221	28
1,1,2,2-Tetrachloroethane	46-157	61
Tetrachloroethene	64-148	39
Toluene	47-150	41
1,1,1-Trichloroethane	52-162	36
1,1,2-Trichloroethane	52-150	45
Trichloroethene	70-157	48
Trichlorofluoromethane	17-181	84
Vinyl chloride	D-251	66

11.3.1.16.4 If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect.

11.3.1.16.5 If in-house QC limits are developed, at least 80% of the analytes tested in the MS/MSD must have in-house QC acceptance criteria that are tighter than those in Section 10.10.1.3 and the remaining analytes (those other than the analytes included in the 80%) must meet the acceptance criteria in Section 11.3.1.1.1. If an in-house QC limit for the RPD is greater than the limit in Section 10.10.1.3, then the limit in the table must be used. Similarly, if an in-house lower limit for recovery is below the lower limit in Section 11.3.1.1.1, then the lower limit in the table must be used, and if an in-house upper limit for recovery is above the upper limit in Section 11.3.1.1.1, then the upper limit in the table must be used.

11.3.1.17 SURROGATE EVALUATION: Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS and MSD. Acceptance criteria can be found in the LIMS: The surrogate recoveries for all QC samples must be within established control limits.

11.3.1.17.1 Method 624.1 – Spike the surrogates into all samples, blanks, LCSs, and MS/MSDs. Compare surrogate recoveries against limits must be developed by the laboratory. In-house QC acceptance criteria must be updated at least every two years. If any recovery fails its criteria, attempt to find, and correct the cause of the failure.

11.3.1.18 INTERNAL STANDARD AREA COUNT: When a calibration is performed at the beginning of an analytical run, the internal standard areas must be evaluated against the mid-point of the curve. Internal standard responses must be -50% to 200% to be acceptable. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve. Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Addition samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV. When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily CCV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

CLIENT NOTE: For Marathon, the internal standard area counts for all calibration standards, QC samples, and samples for quantitation must not change by a factor of greater than (-50% to +130%) as per section 9.2.4

11.3.1.18.1 Method 624.1 – The responses of each internal standard in each sample, blank, and MS/MSD must be within 50% to 200% (1/2 to 2×) of its respective response in the mid-point calibration standard. If, as a group, all internal standards are not within this range, perform and document system repair, repeat the calibration verification/LCS test, and re-analyze the affected samples. If a single internal standard is not within the 50% to 200% range, use an alternative internal standard for quantitation of the analyte referenced to the affected internal standard. It may be necessary to use the data system to calculate a new response factor from calibration data for the alternative internal standard/analyte pair. If an internal standard fails the 50–200% criteria and no analytes are detected in the sample, ignore the failure, or report it if required by the regulatory/control authority.

11.3.1.19 SECOND SOURCE: The second source calibration verification or initial calibration verification standard must be analyzed following each new initial calibration to verify the validity of the calibration standards. The recovery of

the analytes in the SSCV (ICV) must be within 30% of the expected concentration for CCC and SPCC compounds and within 40% for non-CCC/SPCC compounds. Poor performers listed in section 9.2.4.7 must recover within in-house calculated LCS recovery acceptance limits.

STATE NOTE: For all samples analyzed from South Carolina, the SSCV must recover ±30% for all target analytes.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If these criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, or a higher concentration standard can be analyzed. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

11.3.1.19.1 Method 624.1 Calibration verification/LCS—The working calibration curve or RF must be verified immediately after calibration and at the beginning of each 12-hour shift by the measurement of an LCS. The LCS must be from a source different from the source used for calibration, but may be the same as the sample prepared for the DOC.

11.3.1.20 The laboratory participates in semi-annual proficiency testing that meets and/or exceeds the requirements of the Quality Control Sample as listed in the published Standard Method SM 6200B-2011, *Volatile Organic Compounds,* and SM 6020-2011, *Quality Assurance/Quality Control*.

11.3.1.21 For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 12.7.1) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels.

11.3.1.21.1 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).

11.3.1.21.2 The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.

11.3.1.21.3 It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within three (3) years.

11.3.1.21.4 Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

12.3 SITE-SPECIFIC requirements and STATE SPECIFIC criteria must be reviewed and used, if known, for data review.

12.4 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.

12.5 Tune Check - A successful BFB tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria detailed above, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called “Autotunes” that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.

12.6 INITIAL AND CONTINUING CALIBRATION VERIFICATION STANDARD: An Initial Calibration Verification (ICV) standard is analyzed before sample analysis can begin and a continuing calibration verification (CCV) standard was analyzed every 12 hours and meets the criteria in Section 9.0. If these criteria are exceeded and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.

Method 8000D: To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 10.2.4). Percent error between the calculated and expected amounts of an analyte must be $\leq$30% for all standards. For some data uses, $\leq$50% may be acceptable for the lowest calibration point.

12.7 METHOD BLANK: Blank contamination above the report limit – All samples containing detectable amounts above the reporting limit must be re-analyzed or qualified. Samples with no detectable amounts above the reporting limit do not require re-analysis, but the samples must be qualified with blank contamination and it must be mentioned in the case narrative in the data package.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.

- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

Instrument blanks may be injected at any time in the sequence to verify absence of contamination. The source of contamination must be investigated and reduced or eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

12.7.1 Method 8000D: When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

12.8 LABORATORY CONTROL SAMPLES - If the recovery does not meet criteria, see section 12.9 for marginal failures. If it is still out of control limits, then all field and QC samples in the batch must be re-analyzed.

Qualifiers must be applied to any LCS compound that does not meet these criteria and are considered out of control. The percent difference for all method target analytes must be within QC RPD limits. If not, re-analyze the duplicate(s) or prepare a new calibration curve, as necessary.

STATE NOTE: SOUTH CAROLINA DHEC Compliance LCS: responses must be within 70 – 130% for Method 8260 and within the limits given in Appendix F for Method 624. Qualifiers cannot be used. (If an LCS standard is above the acceptable QC criteria and all samples being reported are below the reporting limit, the data is acceptable based on a high bias with undetectable levels in the field samples. Any positive samples require reanalysis.) Failures require a batch re-analysis. For samples analyzed

from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

12.9 LCS/LCSD & MS/MSD CRITERIA

12.9.1 Quality control criteria must be checked for the LCS and LCSD.

LCS samples that do not pass the acceptable QC criteria must be re-analyzed. LCS failures can meet marginal exceedance criteria below. Normal compound list for 8260/624.1 contains typical 90 analytes; therefore, only five analytes can be considered as marginal exceedances. If the failure persists, re-prepare and re-analyze the entire sample batch.

When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside of control limits. Upper and lower marginal exceedance (ME) limits are established by +/- four times the standard deviation. The number of marginal exceedance is based on the number of analytes in the LCS.

Number of allowable marginal exceedances:

>90 analytes, 5 analytes allowed in the ME limit
71 – 90 analytes, 4 analytes allowed in the ME limit
51 – 70 analytes, 3 analytes allowed in the ME limit
31 – 50 analytes, 2 analytes allowed in the ME limit
11 – 30 analytes, 1 analyte allowed in the ME limit
< 11 analytes, no analyte allowed in the ME limit

Marginal exceedances must be random events.

STATE NOTE: For South Carolina DHEC compliance samples, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

12.9.2 **Method 8000D:** If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than five (5) times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.

NOTE: Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

12.10 MATRIX SPIKE ASSESSMENT: If acceptance criteria are not met, perform the following corrective actions as appropriate.

If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-analyzed.
If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.
If analyst error appears to be the root cause of the failure (i.e., no spiked) reanalysis of the MS/MSD and parent is required if volume permits.

Acceptance criteria are that all RPD results must be within the current control limits found in the LIMS. If these conditions are not met, perform the following corrective actions as appropriate.

Re-analyze the sample to verify a matrix effect only if analyst error appears to be the root cause.
If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified.

STATE NOTE: South Carolina DHEC compliance analyses require that all target compounds meet the established MS/MSD criteria. No qualifiers can be applied, except in the circumstance where matrix interference is apparent.

PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples): Acceptance criteria are that all %Recovery and/or RPD results meet project-established goals.

12.11 SURROGATE EVALUATION: If the surrogate recoveries are outside limits for Blank, ICV/CCV/SSCV, and LCS/LCSD, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary. All samples associated with batch or sequence needs to be re-analyzed. The surrogate recoveries for all field samples must be within established control limits. If more than two surrogate recoveries are outside limits, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary which includes qualifying

data with J1 (outside upper limit) or J2 (outside lower limit). When surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).

12.12 INTERNAL STANDARD AREA COUNT: If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 9.2.4, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.

- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
- If any internal standard fails high (> +100%), sample must be re-analyzed with possible dilution. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
- If more than two internal standard fails low « -50%), sample must be re-analyzed. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).
- If one or two internal standard criteria fails low « -50%), corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).

12.13 CALIBRATION RANGE: The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

12.14 SECOND SOURCE (SSCV) or Initial Calibration Verification (ICV): If the SSCV does not meet acceptance criteria, if can be reanalyzed once. If the failure persists, a new initial calibration curve must be prepared and analyzed.

12.15 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

12.15.1 If a method blank contains an amount of target analyte, but all samples are non-detected or the samples contain analyte at a level that is greater than 10 times the level present in the blank, the data is reported with the appropriate “B” flag.

12.15.1.1 When comparing analyte contamination in the blank to possible analyte contamination in the field sample, utilize the sample concentration without applying the multiplier value unless the same multiplier has been applied to the quantitation of the target analytes in the blank.

12.15.2 If the sample surrogate is above the acceptable QC range, but the samples are non-detected for all target analytes, flag the sample with a J1 and report. If the surrogate is below the acceptable QC range, re-analyze the sample if the surrogate still fails, re-extract and re-analyze or flag data.

12.15.3 Matrix spike failures must be flagged with "J5" (high) or "J6" (low), when QC limits are exceeded. If there is an RPD failure, the data is flagged with a “J3”.

12.16 Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in ENV-SOP-CORQ-0006, *Manual Integration*. “Before” and “After”

quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.

12.17 Data must be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high calibration standard. Values falling above the high standard must be diluted and re-analyzed.

12.17.1 Site specific DQO's may require values below the reporting limit but above the method detection limit be reported as "UJ" or estimated value. The reporting limit is the concentration of the lowest standard used in the calibration.

12.17.2 All tentatively identified compounds (TICs) are reported with a "J" qualifier for estimated value and an "N" for presumptive evidence of material.

12.18 For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

13.1 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ENV-SOP-MTJL-0051, Waste Management Plan.*

13.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

14.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

14.3 Heated purge may be used for all samples regardless of matrix or method.

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15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Appendix A: Target Analyte List and Routine LOQ

16.2 Appendix B: QC Summary

16.3 Appendix C: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from SW-846 Method 8260B Table 5

16.4 Appendix D: Potential Compounds to be Analyzed by this Procedure

16.5 Appendix E: The SIM Mode

16.6 Appendix F: EPA 624.1 CCC Criteria

16.7 Appendix G: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

16.8 Appendix H: EPA 8260D (Table 4) Guidance Response Factors Criteria From EPA Contract Laboratory Program (Min RF

16.9 Appendix I: Laboratory Control Standard and Matrix Spike Typically Spiked Compounds

16.10 Appendix J: DoD Requirements

17.0 REFERENCES

17.1 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.

17.2 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.

17.3 *Determinative Chromatographic Separations*, SW-846 Method 8000D, Revision 4, July 2014.

17.4 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260B, Revision 2, December 1996.

17.5 *Purgeables*, 40 CFR 136, EPA Method 624/

17.6 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method*, SM 6200B, 20th edition.

17.7 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference

17.8 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260C, Revision 3, August 2006.

17.9 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method*, SM 6200B-2011.

17.10 *Purgeables by GC/MS*, EPA Method 624.1, Federal Register, Volume 82, Number 165, August 28, 2017.

17.11 40 Code of Federal Regulations §136.6(b)(4)(j).

17.12 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry,* SW-846 Method 8260D, Revision 4, June 2018.

18.0 Revision history

This Version:

Section	Description of Change
All	Complete SOP reformat.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MTJL-0100	Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)	04

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Appendix A: Target Analyte List and Routine LOQ

Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
1,1,1,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,3-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,3-Trichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dibromo-3-Chloropropane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2-Dibromoethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3,5-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Butadiene	0.002	mg/L	0.002	mg/Kg	0.125	mg/Kg
1,3-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,4-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2,4-Trimethyl Pentane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2-Butanone (MEK)	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
2-Chloroethyl vinyl ether	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
2-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Ethyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Methyl-2-pentanone (MIBK)	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Acetone	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Acrolein	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Acrylonitrile	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Benzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromodichloromethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromoform	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromomethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Carbon tetrachloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorodibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
Chloroform	0.005	mg/L	0.005	mg/Kg	0.1	mg/Kg
Chloromethane	0.0025	mg/L	0.0025	mg/Kg	0.05	mg/Kg
cis-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
cis-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dichlorodifluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Dicyclopentadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Ethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexachlorobutadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexane	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Isopropylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methylene Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Naphthalene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
n-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
n-Propylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
p-Isopropyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Propene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
sec-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Styrene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
tert-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tetrachloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Toluene	0.001	mg/L	0.001	mg/Kg	0.25	mg/Kg
trans-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Vinyl chloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Xylenes, Total	0.003	mg/L	0.003	mg/Kg	0.15	mg/Kg
Additional Compounds						
1,4-Dioxane+	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
2-Butanol	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
2-Hexanone	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
2-Propanol	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Acetonitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Allyl Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Bromoethane	0.01	mg/L	0.001	mg/Kg	0.05	mg/Kg
Carbon Disulfide	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroprene	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Cyclohexanone	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Dichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
Ethanol	0.1	mg/L	0.1	mg/Kg	2.5	mg/Kg
Ethyl methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Ethyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Iodomethane	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Isobutanol	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
Isobutanol	0.1	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methacrylonitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Methyl Methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Pentachloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Propionitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Tert Amyl Alcohol	0.05	mg/L	0.05	mg/Kg	0.25	mg/Kg
Tert Butyl Ethyl Alcohol	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
Tert-Amyl Methyl Ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tert-Butyl Alcohol	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Tert-Butyl Formate	0.02	mg/L	0.02	mg/Kg	1	mg/Kg
Tetrahydrofuran	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Trans-1,4-Dichloro-2-butene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
Vinyl Acetate	0.01	mg/L	0.01	mg/Kg	2.5	mg/Kg

RLs are based on a 5mL purge volume
Low Soil - Using a 5g soil sample to 5mL water – See Method 5035 (ENV-SOP-MTJL-0129) Section 8.2.4.1
High Soil – Using 200uL extract from 10g soil sample to 10mL methanol; see Method 5035 (ENV-SOP-MTJL-0129) Sect. 8.3.1.2
+ 1,4-Dioxane has a RL of .002 when run using the SIM mode.

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Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	At instrument set up, after CCV failure	Must meet one of curve fit options presented in Section 9.0. For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint cal bration standard.	Identify and correct source of problem, repeat	None. Do not proceed with analysis
ICV	After Each ICAL	All analytes must be within ± 30% recovery (%R) of the true value with the exception of poor performers which must be within in-house limits.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
RT Window Position (Daily)	Once per ICAL and at the beginning of the analytical window.	Position is set using the mid-point of the ICAL on the day ICAL is performed; otherwise mid-point of CCV is used	NA	NA
RT Window Study	At method set-up and after major instrument maintenance	Window is ± 3 times the standard deviation among three data points across 72 hours.	NA	NA
CCV	Daily, before sample analysis, after every X, and at end of analytical window.	Opening CCV: All analytes within ± 20 %D Ending CCV: All analytes within ± 50 %D	See Section 12 for required corrective actions based on circumstance.	Qualify analytes with CCV out of of criteria.
Internal Standards	Every field sample, standard and QC sample	Must meet criteria specified in Section 9.0.	Troubleshoot instrument performance. Reanalyze samples.	Qualify outages and explain in case narrative.
Surrogate	All field and QC samples.	Routine limits are presented in the LIMS	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.

Method Blank	One per preparatory batch.	No analytes detected above the MDL for any of the method target analytes	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS	One per preparatory batch.	Routine LCS control limits are presented in the LIMS	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.
MS/MSD	One per preparatory batch.	All RPD results must be within the indicated control limits found in the LIMS.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.
Trip Blank	1 per cooler	No analytes detected above the MDL for any of the method target analytes	Examine the project specific requirements. Contact the client as to additional measures to be taken.	NA
Tune Standard	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.
Performance Check	Prior to ICAL	Must meet the minimum, average RF criteria for each analyte according to Section 9.2.3.2.4-9.2.3.2.7	Identify source of problem and troubleshoot instrument performance. Repeat ICAL.	None. Do not proceed with analysis

Appendix C: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from SW-846 Method 8260B Table 5

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
1,3-Butadiene	39	54
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Dicyclopentadiene	66	132
1,2-Dibromo-3-chloropropane	157**	75, 155
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d4	152	115, 150
1,3-Dichlorobenzene	146	111, 148

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
4-Ethyltoluene	105	120
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
Hexane	57	86, 56
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53

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Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Propene	41	39, 42
Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzen	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tert-butyl formate	59	57, 41
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
1,4-Difluorobenzene	114	63
1,4-Dichlorobenzene-d4	152	115, 150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
Dichloroethane-d4	102	
Toluene-d8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).
** Primary ion modified due to coelution.

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Appendix D: Potential Compounds to be Analyzed by this Procedure

Acetone
Acetonitrile
Acrolein
Acrylonitrile
Allyl alcohol
Allyl chloride
Benzene
Benzyl chloride
Bromoacetone
Bromochloromethane (I.S.)
Bromodichloromethane
4-Bromofluorobenzene (Surr.)
Bromoform
Bromomethane
2-Butanone
Carbon disulfide
Carbon tetrachloride
Chloral hydrate
Chlorobenzene
Chlorobenzene d-5 (I.S.)
Chlorodibromomethane
2-Propanol
Chloroethane
2-Chloroethanol
bis-(2-Chloroethyl) sulfide
2-Chloroethyl vinyl ether
Chlorofrom
Chloromethane
Chloroprene
3-Chloropropionitrile
1,2- Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
1,4-Dichloro-2-butene
dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane d-4 (surr.)
1,1-Dichloroethene
Trans-1,2-dichloroethene
Cis-1,2-dichloroethene
1,2-dichloropropane
Dicyclopentadiene
1,4-Dioxane
Epichlorohydrin
Ethanol
Ethylbenzene
Ethylene oxide
Ethyl methacrylate
n-Hexane
2-Hexanone
2-Hydroxypropionitrile
Iodomethane
Isobutylalcohol
Malononitrile
Methacrylonitrile
Methylene chloride
Methyl iodide
Methyl methacrylate
4-methyl-2-pentanone
Pentachloroethane
2-Picoline
Propargyl alcohol
Propene
B-propiolactone
Propionitrile
n-Propylamine
Pyridine
Styrene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,3-Butadiene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl acetate
Vinyl chloride
Xylene (total)
1,2,3,4-Diepoxybutane
4-Ethyltoluene

Appendix E: The SIM Mode:

An alternate way of running compounds to achieve lower detection limits is by way of the Single Ion Monitoring (SIM) method. The SIM method allows the Mass spec to dwell on certain ions rather than scanning the full range of masses from 35 to 300. This process allows for much lower detection limit of desired compounds. This method is only for the detection of known compounds while a TIC cannot be performed while running the SIM method. Currently 1,4-Dioxane is the only compound that is analyzed using the SIM method in the volatiles laboratory.

Appendix F: EPA 624.1 CCC Criteria:

Table 7 – LCS (Q), DOC (s and $\bar{X}$), and MS/MSD (P and RPD) Acceptance Criteria[1]

Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P_1, P_2 (%)	Limit for RPD
Acrolein	60-140	30	50-150	40-160	60
Acrylonitrile	60-140	30	50-150	40-160	60
Benzene	65-135	33	75-125	37-151	61
Benzene-d_6					
Bromodichloromethane	65-135	34	50-140	35-155	56
Bromoform	70-130	25	57-156	45-169	42
Bromomethane	15-185	90	D-206	D-242	61
2-Butanone-d_5					
Carbon tetrachloride	70-130	26	65-125	70-140	41
Chlorobenzene	65-135	29	82-137	37-160	53
Chloroethane	40-160	47	42-202	14-230	78
Chloroethane-d_5					
2-Chloroethylvinyl ether	D-225	130	D-252	D-305	71
Chloroform	70-135	32	68-121	51-138	54
Chloroform-^{13}C					
Chloromethane	D-205	472	D-230	D-273	60
Dibromochloromethane	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	65-135	31	59-174	18-190	57
1,2-Dichlorobenzene-d_4					
1,3-Dichlorobenzene	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	65-135	31	59-174	18-190	57
1,1-Dichloroethane	70-130	24	71-143	59-155	40
1,2-Dichloroethane	70-130	29	72-137	49-155	49
1,2-Dichloroethane-d_4					
1,1-Dichloroethene	50-150	40	19-212	D-234	32
1,1-Dichloroethene-d_2					
trans-1,2-Dichloroethene	70-130	27	68-143	54-156	45
1,2-Dichloropropane	35-165	69	19-181	D-210	55
1,2-Dichloropropane-d_6					
cis-1,3-Dichloropropene	25-175	79	5-195	D-227	58
trans-1,3-Dichloropropene	50-150	52	38-162	17-183	86
trans-1,3-Dichloropropene-d_4.					
Ethyl benzene	60-140	34	75-134	37-162	63
2-Hexanone-d_5					
Methylene chloride	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	60-140	36	68-136	46-157	61
1,1,2,2-Tetrachloroethane-d_2					
Tetrachloroethene	70-130	23	65-133	64-148	39
Toluene	70-130	22	75-134	47-150	41
Toluene-d_8					
1,1,1-Trichloroethane	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	70-130	27	75-136	52-150	45
Trichloroethene	65-135	29	75-138	70-157	48
Trichlorofluoromethane	50-150	50	45-158	17-181	84
Vinyl chloride	5-195	100	D-218	D-251	66
Vinyl chloride-d_3					

[1] Criteria were calculated using an LCS concentration of 20 µg/L

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Appendix G: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification:

Volatile Compound	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100

Volatile Compound	Minimum Response Factor (RF)
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Appendix H: EPA 8260D (Table 4) Guidance Response Factors Criteria From EPA Contract Laboratory Program (Min RF):

Analyte	RF
Acetone	0.01
Benzene	0.2
Bromochloromethane	0.1
Bromodichloromethane	0.3
Bromoform	0.1
Bromomethane	0.01
2-Butanone	0.01
Carbon disulfide	0.1
Carbon tetrachloride	0.1
Chlorobenzene	0.4
Chloroethane	0.01
Chloroform	0.3
Chloromethane	0.01
Cyclohexane	0.01
Dibromochloromethane	0.2
1,2-Dibromo-3-chloropropane	0.01
1,2-Dibromoethane (EDB, Ethylene dibromide)	0.2
1,2-Dichlorobenzene	0.6
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.6
Dichlorodifluoromethane	0.01
1,1-Dichloroethane	0.3
1,2-Dichloroethane	0.07
1,1-Dichloroethene (Vinylidene chloride)	0.06
cis-1,2-Dichloroethene	0.2
trans-1,2-Dichloroethene	0.1
1,2-Dichloropropane	0.2
cis-1,3-Dichloropropene	0.3
trans-1,3-Dichloropropene	0.3
Ethylbenzene	0.4
2-Hexanone	0.01
Isopropylbenzene	0.4
Methyl acetate	0.01
4-Methyl-2-pentanone	0.03
Methyl tert-butyl ether (MTBE)	0.1
Methylcyclohexane	0.05
Methylene chloride	0.01
Styrene	0.2
1,1,2,2-Tetrachloroethane	0.2
Tetrachloroethene	0.1
Toluene	0.3
1,2,3-Trichlorobenzene	0.4
1,2,4-Trichlorobenzene	0.4
1,1,1-Trichloroethane	0.05
1,1,2-Trichloroethane	0.2
1,1,2-Trichlorotrifluoroethane	0.05
Trichloroethene (Trichloroethylene)	0.2
Trichlorofluoromethane	0.01
Vinyl chloride	0.01
m,p-Xylene	0.2
o-Xylene	0.2

Values in this table are referenced from the CLP Statement of Work SOM 02.4. These response factors are provided as guidance only and are not intended to be a requirement. See Appendix B for additional information.

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Appendix I: Laboratory Control Standard and Matrix Spike Typically Spiked Compounds

STANDARD ANALYTE LIST

1,1,1,2-TETRACHLOROETHANE
1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1,2-TRICHLOROTRIFLUOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROPROPENE
1,2,3-TRICHLOROBENZENE
1,2,3-TRICHLOROPROPANE
1,2,3-TRIMETHYLBENZENE
1,2,4-TRICHLOROBENZENE
1,2,4-TRIMETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE
1,2-DIBROMOETHANE
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3,5-TRICHLOROBENZENE
1,3,5-TRIMETHYLBENZENE
1,3-BUTADIENE
1,3-DICHLOROBENZENE
1,3-DICHLOROPROPANE
1,4-DICHLOROBENZENE
1-METHYLNAPHTHALENE
2,2,4-TRIMETHYLPENTANE
2,2-DICHLOROPROPANE
2-BUTANONE (MEK)
2-CHLOROETHYL VINYL ETHER
2-CHLOROTOLUENE
2-HEXANONE
2-METHYLNAPHTHALENE
4-CHLOROTOLUENE
4-ETHYLTOLUENE
4-METHYL-2-PENTANONE (MIBK)
ACETONE
ACROLEIN*ACRYLONITRILE*
CHLORODIBROMOMETHANE
CHLOROETHANE
CHLOROFORM
CHLOROMETHANE
CIS-1,2-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
DIBROMOMETHANE
DICHLORODIFLUOROMETHANE
DICHLOROFLUOROMETHANE
DICYCLOPENTADIENE
DI-ISOPROPYL ETHER
ETHYL ETHER
ETHYLBENZENE
HEXACHLORO-1,3-BUTADIENE
IODOMETHANE
ISOPROPYLBENZENE
M&P-XYLENE
METHYL TERT-BUTYL ETHER
METHYLENE CHLORIDE
NAPHTHALENE
N-BUTYLBENZENE
N-HEXANE
N-PROPYLBENZENE
O-XYLENE
P-ISOPROPYLTOLUENE
PROPENE
SEC-BUTYLBENZENE
STYRENE
TERT-BUTYLBENZENE
TETRACHLOROETHENE
TETRAHYDROFURAN
TOLUENE
TPH (GC/MS) LOW FRACTION
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
TRANS-1,4-DICHLORO-2-BUTENE
TRICHLOROETHENE

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STANDARD ANALYTE LIST

ACRYLONITRILE*BENZENE
BENZENEBROMOBENZENE
BROMOBENZENE
BROMOCHLOROMETHANE
BROMODICHLOROMETHANE
BROMOFORM
BROMOMETHANE
CARBON DISULFIDE
CARBON TETRACHLORIDE
CHLOROBENZENE
TRICHLOROFLUOROMETHANE
VINYL ACETATE
VINYL BROMIDE
VINYL CHLORIDE
XYLENES, TOTAL

SURROGATE LIMITS

4-BROMOFLUOROBENZENE
2,2-DICHLOROETHANE-D4
TOLUENE-D8

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Appendix J: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

3.0 Troubleshooting

Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible, reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes

Table 1. GCMS Troubleshooting Guide

Problem	Cause	Treatment
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseat compression fitting
No tune peaks	Cal gas valve not open	Open cal gas valve
	PFTBA solenoid valve stuck open. All PFTBA has evaporated.	Have solenoid replaced. Put fresh PFTBA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	"Blown" septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.

4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used, or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e., 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.7 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the

LOD/LOQ verifications on the worst-case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least two (2) times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- # The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- # If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- # The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- # The DL and LOD must be reported for all analyte-matrix-methods suites unless it is not applicable to the test or specifically excluded by project requirements.

4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- # If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This

approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

- # Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- # If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- # Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- # Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.19 Surrogate spike results shall be compared with DoD LCS limits (see Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.20 The method blank shall be considered to be contaminated if:

- # The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater.
- # The concentration of any common laboratory contaminant in the blank exceeds the LOQ.
- # If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	11115	101.1	7.8	78	125
71-55-6	1,1,1-Trichloroethane	12156	101.6	9.4	73	130
79-34-5	1,1,2,2-Tetrachloroethane	11670	97	8.9	70	124
79-00-5	1,1,2-Trichloroethane	11772	99.7	7.2	78	121
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	9760	100.8	11.7	66	136
75-34-3	1,1-Dichloroethane	11856	100.4	8.1	76	125
75-35-4	1,1-Dichloroethene	12352	100.3	10.1	70	131
563-58-6	1,1-Dichloropropene	10793	100.5	8.3	76	125
87-61-6	1,2,3-Trichlorobenzene	10572	97.8	10.6	66	130
96-18-4	1,2,3-Trichloropropane	10925	99.1	8.8	73	125
526-73-8	1,2,3-Trimethylbenzene	1948	99.8	6	82	118
120-82-1	1,2,4-Trichlorobenzene	10980	98	10.4	67	129
95-63-6	1,2,4-Trimethylbenzene	11085	98.7	7.9	75	123
96-12-8	1,2-Dibromo-3-chloropropane	11380	96.6	11.7	61	132
106-93-4	1,2-Dibromoethane	11408	100.1	7.3	78	122
95-50-1	1,2-Dichlorobenzene	11785	99.1	7.2	78	121
107-06-2	1,2-Dichloroethane	12328	100.5	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	5951	103.1	10.8	71	136
540-59-0	1,2-Dichloroethene	7748	99.9	7.3	78	122
78-87-5	1,2-Dichloropropane	12145	99.5	7.8	76	123
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	1269	97.8	11.3	64	132
108-70-3	1,3,5-Trichlorobenzene	4723	99.4	9.6	71	128
108-67-8	1,3,5-Trimethylbenzene	11080	98.4	8.4	73	124
541-73-1	1,3-Dichlorobenzene	11619	98.9	7.4	77	121
142-28-9	1,3-Dichloropropane	10713	99.1	7.3	77	121
542-75-6	1,3-Dichloropropene	3714	101.6	8.1	77	126
106-46-7	1,4-Dichlorobenzene	11848	97.5	7.6	75	120
105-05-5	1,4-Diethylbenzene	1896	96.6	5.9	79	114
123-91-1	1,4-Dioxane	7698	96.4	13.7	55	138
544-10-5	1-Chlorohexane	2543	100.4	9.8	71	130
594-20-7	2,2-Dichloropropane	10703	99.7	11.1	67	133
78-93-3	2-Butanone [MEK]	11514	99.6	16.3	51	148
126-99-8	2-Chloro-1,3-butadiene	6667	99	11.3	65	133
110-75-8	2-Chloroethyl vinyl ether	6957	96.1	17.6	43	149
95-49-8	2-Chlorotoluene	10838	98.5	7.9	75	122
591-78-6	2-Hexanone	11004	99.1	15.4	53	145
79-46-9	2-Nitropropane	4969	98.3	17.1	47	150
67-63-0	2-Propanol [Isopropyl alcohol]	1696	99.8	13.4	60	140
460-00-4	4-Bromofluorobenzene	6267	98.9	6.8	79	119
106-43-4	4-Chlorotoluene	10785	98.3	8.6	72	124
108-10-1	4-Methyl-2-pentanone [MIBK]	11364	99.6	11.6	65	135
67-64-1	Acetone	11089	99.6	21.4	36	164
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-43-4	Dichlorofluoromethane	717	100.8	18	47	155
60-29-7	Diethyl ether	6283	99.6	9.6	71	129
108-20-3	Diisopropyl ether	8542	98.3	9.7	69	127
64-17-5	Ethanol	3958	102.2	18.9	45	159
141-78-6	Ethyl acetate	4516	95.4	14.5	52	139
97-63-2	Ethyl methacrylate	7075	98.9	9.9	69	129
637-92-3	Ethyl tert-butyl ether	7514	98.9	9.1	72	126
100-41-4	Ethylbenzene	12427	99.1	7.7	76	122
462-06-6	Fluorobenzene	689	97.3	5.4	81	114
142-82-5	Heptane	5420	93.4	14.9	49	138
87-68-3	Hexachlorobutadiene	10264	98.1	12.4	61	135
67-72-1	Hexachloroethane	3265	102.5	10.1	72	133
110-54-3	Hexane	7116	93.6	16.1	45	142
74-88-4	Iodomethane	9457	100.9	10.1	71	131
78-83-1	Isobutyl alcohol	6162	97.5	12.6	60	135
108-21-4	Isopropyl acetate [Acetic acid]	2885	94.2	12.2	58	131
98-82-8	Isopropylbenzene	11596	100.8	11.1	68	134
179601-23-1	m/p-Xylene [3/4-Xylene]	10612	100.4	7.7	77	124
126-98-7	Methacrylonitrile	6736	99.2	11.1	66	132
79-20-9	Methyl acetate	8320	98.7	15.2	53	144
80-62-6	Methyl methacrylate	7050	98.4	11.9	63	134
1634-04-4	Methyl tert-butyl ether [MTBE]	11253	98.9	8.7	73	125
108-87-2	Methylcyclohexane	8565	99.4	11.2	66	133
75-09-2	Methylene chloride	12024	98.9	9.7	70	128
123-86-4	n-Butyl acetate	2981	95.1	11	62	128
71-36-3	n-Butyl alcohol	4800	92.9	12.6	55	131
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125

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Table 3. LCS Control Limits – Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	24511	101.1	7.6	78	124
71-55-6	1,1,1-Trichloroethane	28223	102.7	9.6	74	131
79-34-5	1,1,2,2-Tetrachloroethane	27450	96.4	8.3	71	121
79-00-5	1,1,2-Trichloroethane	27338	99.5	6.5	80	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	21122	103	11.1	70	136
75-34-3	1,1-Dichloroethane	28154	101.3	8	77	125
75-35-4	1,1-Dichloroethene	29436	101	10	71	131
563-58-6	1,1-Dichloropropene	23631	102	7.8	79	125
87-61-6	1,2,3-Trichlorobenzene	24271	98.7	10.1	69	129
96-18-4	1,2,3-Trichloropropane	24525	97.5	8	73	122
526-73-8	1,2,3-Trimethylbenzene	2965	100.9	6.2	82	120
120-82-1	1,2,4-Trichlorobenzene	25290	99.8	10.1	69	130
95-63-6	1,2,4-Trimethylbenzene	27917	99.6	8	76	124
96-12-8	1,2-Dibromo-3-chloropropane	24955	94.9	11.1	62	128
106-93-4	1,2-Dibromoethane	29096	99	7.2	77	121
95-50-1	1,2-Dichlorobenzene	27583	99.4	6.5	80	119
107-06-2	1,2-Dichloroethane	32965	100.3	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	8673	99.5	6.1	81	118
540-59-0	1,2-Dichloroethene	18667	100.2	7.1	79	121
78-87-5	1,2-Dichloropropane	27787	100.1	7.2	78	122
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	3144	103.1	10.9	70	136
108-70-3	1,3,5-Trichlorobenzene	10037	102.1	9.2	75	130
108-67-8	1,3,5-Trimethylbenzene	27820	99.5	8.1	75	124
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129

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CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
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75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
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74-87-3	Chloromethane	27697	94.5	15	50	139
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74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116

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CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128

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CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane	26108	103	12.8	65	141
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127

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110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane [Freon-11]	26108	103	12.8	65	141
108-05-4	Vinyl acetate	18941	100.2	15.3	54	146
75-01-4	Vinyl chloride	29472	97.4	13.2	58	137
1330-20-7	Xylenes [total]	23426	100.1	7	79	121

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression for each analyte: $r^2 \geq 0.99$; **Option 3**: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL s performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat the CCV and all associated samples since the last successful CCV. Alternately, Recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 and 4 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Document Information

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Signature Manifest

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QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality	17 Feb 2022, 11:42:57 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Supervisor	14 Dec 2021, 03:29:55 PM	Approved
[redacted]	Quality Analyst 3	17 Dec 2021, 12:44:45 PM	Approved

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Multi-Increment Sampling
TEST METHOD: NA
ISSUER: Pace Analytical National Center for Testing & Innovation

1.0 Scope and Application

1.1 Appendix A of EPA Method 8330B (SW-846) specifically addresses field sampling. The appendix provides guidance for explosive residue sample collection, handling, and laboratory processing techniques. Method 8330B recommends the use of multi-increment (MI) sampling, which involves the extraction of a representative portion of material from within a single decision unit which will adequately address potential compositional and distributional heterogeneity. In MI sampling, several increments from the same decision unit are combined to form one sample that is submitted for laboratory analysis. The procedures for MI sampling are specifically designed to minimize sampling error and provide a more scientifically-representative mean concentration of the contaminant(s) present in the decision unit.

1.2 Initial demonstration for achieving samples size below 75µm per DOD/DOE QSM is on file in the QA department.

2.0 Summary of Method

2.1 Samples are dried, ground, and homogenized before subsamples are taken for sample preparation.

3.0 Interferences

3.1 Care must be taken to not cross-contaminate samples during the drying, sieving, and grinding procedures. Grinding blanks are required to verify procedure is free from cross contamination.

3.2 The drying process may result in quantitative losses of some analytes. Project Managers may consider eliminating the drying process prior to analysis or removing poor performers from the target analyte list if drying is required.

4.0 Definitions

4.1 Sieve: A device made of wire mesh held in a frame through which finer particles of a mixture of various sizes may be passed to separate them from coarser ones or through which soft materials may be forced for reduction to fine particles.

4.2 Shatterbox: A device for mechanically pulverizing a sample or material.

4.3 Ball Mill: A device using ceramic pellets and rotation in a closed container to pulverize the contents.

4.4 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

5.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

5.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

5.3 Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

5.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

5.5 Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 Sample Collection, Preservation, Holding Time, and Storage

6.1 Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

6.2 Pace National will typically receive samples in 4-8oz containers for processing.

7.0 Equipment and Supplies

7.1 Sieve: 10mesh

7.2 Grinder: Shatterbox or equivalent capable of reducing particle size to <75µm

7.3 Drying rack

7.4 12-inch brass pans

7.5 Aluminum baking sheets

8.0 Reagents and Standards

8.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted.

9.0 Procedure

9.1 All sample contents within the container are emptied into a pan/weigh boat and dried to a constant weight.

9.1.1 A Blank matrix must be dried with samples.

9.1.2 Obtain a clean pan/weigh boat and record the tare weight.

9.1.3 Empty the entire contents of the sample container into the pan/weigh boat.

9.1.4 Using gloved hands break the soil into small pieces as necessary to facilitate the drying process. Use fresh gloves for each sample to prevent cross contamination.

9.1.5 Record the initial weight of the entire sample.

9.1.6 After the initial weight is obtained, dry the sample at room temperature in a hood for approximately 24 hours. Then obtain a 2nd sample weight.

9.1.7 Continue the drying process for approximately 12 hours and obtain a 3rd sample weight.

9.1.8 Two consecutive weights of less than 10% difference, taken approximately 12 hours apart, is considered to be dried to a constant weight.

9.1.9 Dates/Times are recorded as well as the ambient temperature with each weighing of samples.

9.2 For all methods or when client-specific data quality objectives (DQOs) require grinding, dried sample is introduced into the shatterbox or equivalent. The entire sample must be ground. If multiple portions are ground separately, the aliquots must be combined prior to subsampling for extraction. Samples are ground up to three-minute intervals. Intervals and duration are dependent on the sample matrix and analytes of interest for the specific project. The Blank and weekly check sample must also proceed through this step.

9.3 Dried sample material is passed through a 10mesh (2mm) sieve (may be assisted using gloved hands). Do not intentionally include vegetation unless project specifications include this requirement. Depending on sample matrix, sieving may be performed initially to facilitate the drying process.

9.4 The Blank matrix is ground at the end of each batch. A blank will also be ground after any sample of known concentration above detectable limits, including quality control samples.

9.5 Each sample/QC is spread into a pan in order to perform sufficient subsampling of the final sample aliquot. At least 30 sample increments must be taken for the subsampling procedure. The sample volume extracted for analysis should represent the entire ground sample.

NOTE: If sample volume does not allow 30 aliquots, a note will be made on the extraction log.

9.6 See the specific method extraction SOP for further processing information.

10.0 Data Analysis and Calculations

10.1 See the Laboratory Quality Assurance Manual for equations for common calculations.

11.0 Quality Control and Method Performance

11.1 Analyst Qualifications and Training

11.1.1 Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications for Chemistry* for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

12.1.1 Pace National's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

12.1.2 The review steps and checks that occur as employees complete tasks and review their own work is called primary review.

12.1.3 All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace National's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

12.1.4 A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

12.1.5 Refer to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

12.2.1 Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

13.0 Pollution Prevention and Waste Management

13.1 Pace National proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced

solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

13.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace National's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

14.1 Pace National is set up currently to process from 4oz/8oz/16oz/32oz jars that have been prepared in the field from bulk containers. Pace National cannot currently process bulk samples for this method.

14.2 Due to limited sample volume received as listed in 14.1:

14.2.1 Duplicate subsampling is performed rather than triplicate

15.0 Responsibilities

15.1 Pace National employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace National's policy for temporary departure.

15.2 Pace National supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Not applicable to this SOP

17.0 References

17.1 Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC), SW-846 Method 8330B, Revision 2, October 2006, Appendix A.

17.2 Quality Systems Manual (QSM) for Environmental Laboratories, Department of Defense (DoD), Version 5.1, 2017.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Multi-Increment Sampling
TEST METHOD: NA
ISSUER: Pace Analytical National Center for Testing & Innovation

18.0 Revision History

This Version:

Section	Description of Change
7.3, 7.4, 7.5, 9.2. Removed 8.2, 9.6, & 14.3.	Process update and removal of 8330 prep steps.

This document supersedes the following document(s):

Document Number	Title	Version
ESC Lab Sciences SOP #330377	ESC Lab Sciences SOP #330377	1
ESC Lab Sciences SOP #330377	ESC Lab Sciences SOP #330377	2
ESC Lab Sciences SOP #330377	ESC Lab Sciences SOP #330377	3
ENV-SOP-MTJL-0112	Multi-Increment Sampling	01
ENV-SOP-MTJL-0112	Multi-Increment Sampling	02
ENV-SOP-MTJL-0112	Multi-Increment Sampling	03
ENV-SOP-MTJL-0112	Multi-Increment Sampling	04

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MTJL-0187 **Revision:** 05

Document Title: Toxicity Characteristic Leaching Procedure (EPA Method 1311)

Department(s): Metals

Date Information

Effective Date: 22 Feb 2022

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MTJL-0187 **Revision:** 05

Title: Toxicity Characteristic Leaching Procedure (EPA Method 1311)

All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0187

QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality Program	22 Feb 2022, 08:26:59 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality	04 Feb 2022, 03:24:14 PM	Approved

Pace Analytical®
National Center for Testing & Innovation

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: ENV-SOP—MTJL-0187 Toxicity Characteristic Leaching Procedure
TEST METHOD: EPA 1311

ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 Scope And Application

1.1 This method applies to any sample type (solid, liquid, and multi-phasic waste samples).

1.2 The Toxicity Characteristic Leaching Procedure (TCLP) is designed to determine the environmental mobility of both organic and inorganic contaminants present in liquid, solid, and multi-phasic wastes.

1.3 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste or that they are present but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be analyzed.

1.4 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

2.0 Summary of Method

2.1 For liquid wastes (<0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8µm glass fiber filter, is defined as the TCLP extract. All solid determinations must be performed.

2.2 For wastes containing ≥ 0.5% dry solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes.

2.3 Following extraction the liquid extract is separated from the solid phase by filtration through 0.6 to 0.8µm glass fiber filter. If compatible (multiple phases will not form upon combination), the initial liquid phase of the waste is added to the liquid extract and analyzed together. If they are not compatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 Definitions

4.1 TCLP – The Toxicity Characteristic Leaching Procedure is a method designed to imitate the leaching process that occurs in landfills.

4.2 Liquid phase - The liquid component of the sample that passes through the filter during the initial determination of solids present in the field sample.

4.3 Solid phase - The component of the sample that does not pass through the filter during the initial determination of solids present in the field sample.

4.4 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

Pace Analytical®
National Center for Testing & Innovation

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: ENV-SOP—MTJL-0187 Toxicity Characteristic Leaching Procedure
TEST METHOD: EPA 1311

ISSUER: Pace National – Mt. Juliet, Tennessee

5.0 Health and Safety

5.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

5.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

5.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

5.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

5.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

5.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

5.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

6.0 Sample Collection, Preservation, Holding Time, And Storage

6.1 Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

6.2 Sampling

6.2.1 All samples should be collected in glass or Teflon™ jars. Minimally, a 105g aliquot of solid material should be collected for each analysis needed (It is always wise to collect extra sample in the event that additional extract preparation is required).

6.2.2 A separate aliquot is needed for extraction of volatiles and should be collected with minimal headspace.

6.3 Preservation

6.3.1 Preservatives should not be added to the field sample. The sample should be refrigerated at ≤ 6°C (not frozen) unless it causes physical change to the sample (i.e., precipitation).

6.4 Holding times:

	From Field Collection To TCLP Extraction	From TCLP Extraction To Preparative Extraction	From Preparative Extraction To Determinative Analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Hg	180	NA	180	360

7.0 Equipment And Supplies

7.1 Tumbling apparatus capable of rotating the vessel in an end over end fashion at 30 ± 2rpm

7.2 Bottle Extraction Vessel. When the waste is being evaluated using the non-volatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste. It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than PTFE, shall not be used if organics are to be analyzed.

7.3 Zero Headspace Extraction (ZHE) vessel. This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessels have an internal volume of 500-600mL, and are equipped to accommodate a 90-110mm filter.

7.3.1 The devices contain O-rings, which should be replaced frequently.

7.3.2 For the ZHE to be acceptable for use, the piston within the ZHE should move with approximately 15psi of pressure or less. If it takes more pressure to move the piston, the O-rings need to be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses.

7.3.3 The ZHE should be checked for leaks after every extraction. Pressurize the device to 40psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Re-test the device. If leakage problems cannot be solved, the ZHE is unacceptable for TCLP analyses.

7.4 Borosilicate glass fiber filters must be used and have an effective pore size of 0.6 to 0.8um, or equivalent. When evaluating the mobility of metals, filters must be acid washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with DI water. Alternatively, pre-acid washed filters can be purchased. Glass fiber filters are fragile and should be handled with care.

7.5 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. The type of filter holder used depends on the properties of the material to be filtered. These devices shall have a minimum internal volume of 300mL and be equipped to accommodate a minimum filter size of approximately 47mm (filter holders having an internal capacity of 1.5L or greater, and equipped to accommodate a filter approximately 142mm diameter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid containing wastes. All other types of wastes should be filtered using positive pressure filtration.

NOTE: Record "vacuum" in the filtration device column in Prep Data when a vacuum device is used to filter samples.

7.6 Hazard Waste Pressure Filter System – Millipore (Fisher # YT30 142 HM) or equivalent

NOTE: Record the letter associated with the positive pressure vacuum device in the filtration device column in Prep Data when a positive pressure device is used to filter samples.

7.7 Laboratory Balance and Weigh Boats: Any laboratory balance accurate to within ± 0.01 grams may be used.

7.8 pH Meter: The meter must be accurate to ± 0.05 units at 23 ± 2°C.

7.9 200mL beakers used for initial and adjusted pH

7.10 Magnetic stir plate and stir bars

7.11 Hot plate

7.12 Spatulas (wood and metal)

7.13 Mortar/Pestle

7.14 9.5mm and 1.0mm sieve

7.15 ZHE plunger

7.16 Tin snips and hammer

7.17 Homogenizer

7.18 Tedlar® bags

8.0 Reagents And Standards

8.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital

archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted.

8.2 Reagent grade water

8.3 1N HCl made from ACS reagent grade HCl. Slowly add 83mL of concentrated hydrochloric acid to approximately 500mL of reagent water and then adjust to 1 liter with reagent water. Note: This is an exothermic reaction and should be performed carefully. Always add acid to a larger volume of water; never add water to a larger volume of acid.

8.4 6N NaOH made from ACS reagent grade NaOH. Slowly add 240g of NaOH pellets to approximately 800mL of reagent water, stirring continuously (do not add the water to the NaOH pellets). When the pellets have dissolved completely, adjust the volume to 1 liter with reagent water. Do this twice to obtain 2 liters. Note: This is a highly exothermic reaction and should be performed carefully.

8.5 Glacial Acetic Acid - ACS reagent grade

8.6 TCLP Extraction Fluids:

8.6.1 #1 Extraction Fluid: Add about 120 liters of DI water to a 180L drum. Add 1929mL of 6N NaOH and 1026mL of Glacial Acetic Acid. Bring to a volume of 180L with DI water. The pH of this fluid should be 4.93 ± 0.05.

8.6.2 #2 Extraction Fluid: Add about 80L of DI water to a 100L drum. Add 570mL of Glacial Acetic Acid. Bring to a volume to 100L. The pH of this fluid should be 2.88 ± 0.05.

8.6.3 Upon preparation, if the fluid is ±0.05pH units outside of the acceptable range the fluid must be discarded and re-prepared before use. Also, the fluid must be checked daily prior to preparing samples for tumbling and if the pH of the extraction fluid is beyond the acceptable range, the fluid must be discarded and re-prepared before use.

NOTE: Always check the slope of the pH meter and re-calibrate and re-test the solution, if the fluid is initially determined to be outside the method required pH range.

8.7 pH meter calibration buffers at pH values of 2, 4, 7 and 10 are used to calibrate the pH meter daily. Buffers are used fresh daily then discarded. The slope of the pH meter following calibration must be 98-102. The pH 7 buffer is then re-analyzed and the reading must be 7.0 ± 0.05su.

9.0 Procedure

9.1 Preliminary Evaluation – Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. These preliminary evaluations include:

- Determination of the percent solids
- Determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration
- Determination of whether the solid portion of the waste requires particle size reduction

- Determination of which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste.

9.2 Determination Of Percent Solid

9.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration (100% solids), then skip this step and proceed to particle size reduction if needed.

9.2.2 If the sample is liquid, semi-liquid, or contains liquid; determination of percent solids is required.

NOTE: If the sample is oil, the Project Manager must be notified that the EPA recommends that the sample be run for total analysis. If the PM confirms that the sample must be tumbled for TCLP then the TCLP analysts will inform the PM that the case narrative below must be added to the report.

The TCLP test cannot predict the potential for toxic chemicals to leach from oily waste, through soil, to contaminate ground water. This applies to both sanitary landfills and industrial sites. EPA and the American Society of Testing and Materials (ASTM) have formed a work group to develop a site-specific risk assessment model for oily waste. At a minimum, the model will incorporate physical and chemical characteristics of the oily waste and the soil. However, this model is not expected to be approved by EPA for several years. Until EPA approved this site-specific model for oily waste risk assessments, oily waste site assessments should be based on total constituent analysis, not TCLP extract analysis.

9.2.3 Pre-weigh the filter paper and the container that will receive the filtrate.

9.2.4 Assemble the filter holder.

9.2.5 Homogenize the sample in the original container before a portion of the sample is transferred to a secondary container.

9.2.6 Weigh out a sub-sample of the waste (100 gram minimum) and record the weight of the sample plus the weighing container.

9.2.7 Transfer the sample to the filter holder (liquid and solid phases) and spread the sample evenly over the surface of the filter.

NOTE: Vacuum filtration can be used when samples contain <10% solid and for highly granular liquid containing wastes. Pressure filtration is used in any other case.

9.2.8 Weigh and record the weight of the weighing container that contained the subsample of waste. This weight includes the weight of the container plus any sample residue adhered to it.

9.2.9 Slowly apply pressure of 1-10psi to the filter apparatus until gas moves through the filter. If this point is not reached under 10psi, and if no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10psi increments to a maximum of 50psi. After each incremental increase of 10psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any two minute interval, proceed to the next 10psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased

at 50psi (i.e., filtration does not result in any additional filtrate within any two minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

NOTE: Some wastes, such as oils and paints, will contain liquid material that does not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

9.2.10 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Store the filtrate at ≤ 6°C (not frozen) until the TCLP extraction is completed.

9.2.11 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate filled container.

Weight of liquid phase = (Filtrate + Container) – (Container)

9.2.12 Determine the total weight of sample that was filtered by subtracting the container and residue weight from the total weight of the sample filled container.

Weight of sample filtered= (Sample + Container)–(Container + Residue)

9.2.13 Determine Percent Wet Solids:

$$\text{Percent wet solids} = \frac{\text{Weight of sample filtered (9.2.12)} - \text{Weight of filtrate (9.2.11)}}{\text{Weight of waste filtered (9.2.12)}} \times 100$$

9.2.14 If the percent wet solid is ≥ 5%, then proceed to particle size reduction below if needed.

9.2.15 If the percent wet solids is <0.5% and non-volatile analyses (semivolatiles, pesticides, herbicides, or metals) are required, then the filtrate obtained is considered to be the TCLP extract, so it can be processed accordingly.

9.2.16 If the percent wet solids is <0.5% and volatile analysis is required, then proceed to section 8.6 using a fresh aliquot of sample.

9.2.17 If the percent wet solids is ≥0.5% **and** <5% and some liquid is entrapped in the filter, then determine Percent Dry Solids as follows below.

NOTE: If it is obvious that very little liquid is entrapped in the filter, do not dry filter; proceed to Particle Size Determination. Once the solid phase has been oven-dried, it cannot be used for the Particle Size, Extraction Fluid Determinations, or the TCLP Extraction.

NOTE: If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Particle Size Determination. Once the solid phase has been oven-dried, it cannot be used for the Particle Size, Extraction Fluid Determinations, or the TCLP Extraction.

9.2.18 Dry the filter paper and solid phase at 100 ± 20°C to constant weight (two consecutive stable weights with ±1%) in a drying oven.

9.2.19 Determine Percent Dry Solids:

$$\text{Percent Dry Solids} = \frac{\text{Dried filter and solid phase (9.2.18)} - \text{Filter (9.2.3)}}{\text{Waste filtered (9.2.12)}} \times 100$$

9.2.20 If the Percent Dry Solids is <0.5%, and non-volatile analyses (semivolatiles, pesticides, herbicides, or metals) are required, then the filtrate obtained is considered to be the TCLP extract, so it can be processed accordingly.

9.2.21 If the Percent Dry Solids is <0.5% and volatile analysis is required, then proceed to section 9.6 using a fresh aliquot of sample.

9.2.22 If the Percent Dry Solids is ≥0.5% and the sample will be analyzed for non-volatile constituents, return to the beginning of this section and re-filter a fresh portion of the waste to separate the liquid and solid phases. Once the solid phase has been oven-dried it cannot be used for the Particle Size, Extraction Fluid Determinations, or the TCLP extraction.

9.2.23 If the Percent Dry Solids is ≥0.5% and volatile analysis is required, then proceed to section 9.6 using a fresh aliquot of sample.

9.3 Determination of Particle Size Reduction

9.3.1 Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1cm^2, or is smaller than 1cm in its narrowest dimension (i.e., is capable of passing through a 9.5mm (0.375 inch) standard sieve).

9.3.2 If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are being prepared for organic volatiles extraction, special precautions must be taken (see Section 9.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended.

9.3.3 If all the solid phase will obviously pass through the 9.5mm sieve particle size reduction is not needed and go to section 9.4.

NOTE: The sieve should be used as a gauge of particle size only. Do not actively sieve samples.

9.4 Determination of Type of Extraction Fluid

9.4.1 If the percent solids content of the sample is ≥0.5% and if the sample will be extracted for non-volatile analytes, determine the appropriate extraction fluid.

9.4.2 Reduce a subsample of the solid phase (if necessary) to a particle size of approximately 1mm in diameter or less and transfer 5.0g of the solid into a beaker. (If

sample is multiphasic, filter out the solids and weigh out 5.0g of the solid phase into a beaker.)

NOTE: If a sample is not conducive to particle size reduction down to approximately 1mm diameter, then the reasoning and/or the matrix should be recorded. Examples of material which is not conducive to particle size reduction of approximately 1mm diameter includes (but is not limited to) filters, rags, rocks, and oily materials.

9.4.3 Add 96.5mL of reagent water to the beaker, cover with a watch glass, and stir vigorously until the pH stabilizes or for up to five minutes using a magnetic stirrer. Measure and record the pH.

9.4.4 If the pH ≤5.0, extraction fluid #1 is used.

9.4.5 If the pH>5.0, add 3.5mL 1.0N HCl, slurry briefly, cover with a watch glass, heat to 50°C and hold for 10 minutes. Allow the solution cool to room temperature and record the pH. If the pH is ≤5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

9.5 Non-Volatile TCLP Extraction

9.5.1 A minimum sample size of 100 grams (solid and liquid phases) is required. In some cases, a larger sample size may be appropriate, depending on the dry solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern.

NOTE: A smaller sample amount (<100g) is allowed when a sample volume is sent from customer that is <100g and the customer approves the qualification prior to the leaching and allows the lab to proceed with a short sample.

9.5.2 Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required.

9.5.3 If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

9.5.4 If the sample will obviously yield no liquid when subjected to pressure filtration (100% solid), weigh out a subsample of the waste (100g minimum) and proceed to Section 9.5.6.

9.5.5 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device and filtration procedures described in Section 9.2.

9.5.5.1 After liquid/solid phase separation, store the liquid phase at ≤ 6°C (not frozen) until the TCLP extraction is completed.

9.5.5.2 Transfer the solid phase into the extraction vessel and include the filter used for the phase separation.

9.5.6 Determine the amount of extraction fluid needed:

Volume of Extraction Fluid (mL) = 20 × Solid Phase Weight (g)

9.5.7 Slowly add this amount of appropriate extraction fluid to the extractor vessel.

NOTE: The pH of the extraction fluids needs to be verified each day the fluid is used. Close the extractor bottle tightly, secure in rotary extractor device, and rotate at 30 ± 2 rpm for 18 ± 2 hours.

9.5.8 Check and record the rotation speed in the speed of the tumbler (rpm).

9.5.9 Ambient temperature (temperature of room in which extraction is to take place) shall be maintained at 23 ± 2°C during extraction period. Monitor and record the minimum and maximum room temperature of the extraction during the 18 ± 2 hours extraction time.

9.5.10 After the 18 ± 2 hours extraction is complete, remove the samples from the tumbler and allow the samples to settle.

9.5.11 Separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: The blank which was tumbled must be filtered as well.

9.5.12 If the sample contained no initial liquid phase (100% solids), the filtered liquid leachate obtained is defined as the TCLP extract.

9.5.13 If compatible (multiple phases will not result on combination), combine the filtered liquid leachate with the initial liquid phase of the sample. This combined liquid is defined as the TCLP extract.

9.5.14 If the initial liquid phase of the sample is not or may not be compatible with the filtered liquid leachate, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically.

9.5.15 Following collection of the TCLP extract, the pH of the extract is recorded. The preparatory method should be applied as soon as possible. For organic analysis, extracts are refrigerated at ≤6°C (not frozen) until the extraction. For metal analysis, extracts are typically digested right after they are collected. If digestion of the extracts needs to be delayed, then preserve all samples and blanks with nitric acid to pH <2. If the individual phases are to be analyzed separately, determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average calculation found in section 10.

9.6 Volatile TCLP Extraction

9.6.1 Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold to minimize loss of volatiles. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

9.6.2 Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

9.6.3 Assembling the ZHE

9.6.3.1 Place clean O-rings on the piston, top plate, and bottom plate.

9.6.3.2 Place the top plate upside down on a counter. Place one 90mm screen inside the top plate. Insert one 90mm, glass fiber, 0.7µm filter on top of the screen and cover them with a second 90mm screen. Insert an O-ring on top of the screen/filter sandwich.

9.6.3.3 Wet the piston O-rings with reagent water. Place the barrel right side up and force the piston down to the bottom of the barrel, making sure that the wiper blade side is up. If the sample to be extracted contains free liquid; therefore, must be filtered, do not force the piston to the bottom but only partway down so that it can be filtered.

9.6.3.4 For the ZHE to be acceptable for use, the piston within the ZHE should move with approximately 15psi of pressure or less. If it takes more pressure to move the piston, the O-rings need to be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses. The ZHE should be checked for leaks. Pressurize the device to 40psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Re-test the device. If leakage problems cannot be solved, the ZHE is unacceptable for TCLP analyses.

9.6.4 Place the ZHE piston within the body of the ZHE. Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample. Secure the gas inlet/outlet flange onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange aside.

9.6.5 If the waste is 100% solid, weigh out a sub-sample (25 gram maximum) of the waste. Record the weight.

9.6.6 If the waste contains <0.5% dry solids, the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required.

9.6.7 For wastes containing ≥0.5% dry solids, use the percent solids information to determine the sample size to charge in the ZHE. The recommended sample size is as follows:

9.6.7.1 For wastes containing ≥0.5% and <5% solids, weigh out a 500 gram sample of waste and record the weight.

9.6.7.2 For wastes containing ≥5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge the ZHE (g)} = \frac{25}{\text{Percent Solids}} \times 100$$

9.6.7.3 Weigh out a sub-sample of the waste of the appropriate size and record the weight.

9.6.8 If necessary, prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to the appropriate surface area or particle size. Samples and appropriate reduction equipment should be handled cold. Samples should not be exposed to the atmosphere for any more time than is absolutely necessary.

9.6.9 Quantitatively transfer the sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position. Do not attach the extract collection device to the top plate.

9.6.10 Attach a gas line to the gas inlet/outlet valve and, with the liquid inlet/outlet valve open, begin applying gentle pressure of 1-10psi to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

9.6.11 If the sample is 100% solid, slowly increase the pressure to a maximum of 50psi to force the headspace out of the device.

9.6.12 If the sample is <100% solid, then attach a pre-weighed filtrate collection container (Tedlar® bag) to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10psi to force the liquid phase of the sample into the Tedlar® bag. If no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10psi increments to a maximum of 50psi. After each incremental increase of 10psi, if no additional liquid has passed through the filter in any two minute interval, proceed to the next 10psi increment. When liquid flow has ceased such that continued pressure filtration at 50psi does not result in any additional filtrate within a two minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

9.6.13 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

9.6.14 The liquid phase may now be either analyzed immediately or stored at ≤6°C (not frozen) under minimal headspace conditions until time of analysis.

9.6.15 Determine the appropriate amount of Extraction Fluid #1 to add to the ZHE:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{Percent solids} \times W_s}{100}$$

where: W_s = Weight of sample used (liquid and solid phases)

9.6.16 The following sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases for leaching volatile components.

9.6.16.1 With the ZHE in the vertical position, attach a line from the extraction fluid to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston, open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of the fluid has been introduced into the device.

9.6.16.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion two or three times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10psi and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with about 30psi and check all ZHE fittings to ensure that they are closed.

9.6.17 Place the ZHE in the rotary agitation apparatus and rotate at 30 ± 2rpm for 18 ± 2 hours. Ambient temperature must be maintained at 23 ± 2°C during agitation.

9.6.18 Following the 18 ± 2 hour agitation period, check and record the pressure from the ZHE gauge. If the pressure has not been maintained, the device is leaking. Check the ZHE for leaking as specified in Section 7.3, and repeat again with new sample.

STATE NOTE: A specific criteria for the amount of loss is required when preparing and analyzing samples from Wisconsin. If more than 10% (4-5psi) of the charged pressure is lost, the sample must be re-extracted. When significant loss is observed, the O-rings are replaced as a part of the corrective action process.

9.6.19 If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. Filter through the glass fiber filter, using the ZHE device. The sample is filtered into VOA vials using needles to ensure that loss of volatile analytes is minimized.

9.6.20 If the original waste contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract.

9.6.21 If the waste contained an initial liquid phase, the filtered liquid material obtained and the initial liquid phase are collectively defined as the TCLP extract. Recombination – The initial liquid filtered from the sample is kept in a Tedlar® bag for later recombination. The filtrate after rotation is then added to the Tedlar® bag containing the initial liquid filtrate. Once mixed the sample can be transferred to a VOA vial for analysis.

9.6.22 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at ≤6°C (not frozen) until analyzed. Analyze the

TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using the simple volume weighted average equation found in Section 10.

9.7 Spikes - For every sample type, for every parameter being tested there must be sufficient sample for a spike and a duplicate spike. This will be triple the amount that would be normally be provided to analytical personnel. The spike is added at the bench during analytical preparation, not during TCLP extraction.

9.8 Determination of Leachate Needed

Metals - a minimum of 50mL
Mercury - a minimum of 50mL
Pesticides and PCB's - a minimum of 250mL
BNA and PAH - a minimum of 250mL
Herbicides - minimum of 250mL
All volatiles – (1) 40mL vial

STATE NOTE: The State of South Carolina's Bureau of Land & Waste Management may require drinking water detection limits. These low limits may also require a sample volume of 1 liter. For those samples that fail to meet the above criteria for these specific samples a narrative needs to accompany the samples explaining the samples extraction and why it failed to meet the required limits.

9.9 Clean up – All Lab ware must be clean and dry before use. Extraction vessels and volumetric ware are washed in Miele lab dishwashers using the preprogramed wash cycle and a combination of ProCare Lab 10AP Detergent and ProCare Lab 30C Neutralizer or equivalent.

10.0 Data Analysis And Calculations

10.1 Mathematical combination of incompatible liquid phase/leachate:

$$\text{Final Analyte Concentration} = \frac{(V1)(C1)+(V2)(C2)}{V1+V2}$$

where:
V1 = The volume of the first phases (L).
C1 = The concentration of the analyte of concern in the first phase (mg/L).
V2 = The volume of the second phase (L).
C2 = The concentration of the analyte of concern in the second phase (mg/L).

10.2 $\text{Percent Dry Solids} = \frac{\text{Dried filter and solid phase (9.2.18) – Filter (9.2.3)}}{\text{Waste filtered (9.2.12)}} \times 100$

10.3 $\text{Percent wet solids} = \frac{\text{Weight of sample filtered (9.2.12) – Weight of filtrate (9.2.11)}}{\text{Weight of waste filtered (9.2.12)}} \times 100$

10.4 PPF (Percent Pass Filter) = 100 – %Solids

10.5 See the Laboratory Quality Assurance Manual for equations for common calculations.

11.0 Quality Control and Method Performance

11.1 Extraction Batches: Extraction batches are defined as sets of 1 - 20 samples. Extraction batches must include the following: 1 method blank, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair (if enough sample is available) and Duplicate (Dup). Exceptions are made for waste dilution samples where the minimum batch QC must include a blank, and LCS/LCSD pair.

Note: A 1-liter bulk container of leachate fluid is delivered to the VOC department whenever a new batch of leachate fluid is made. This is to provide extra leachate for making batch blanks and LCSs.

11.2 A minimum of one blank (using the same extraction fluid as used for the samples) for every 20 extractions that have been conducted in an extraction vessel.

NOTE: Be sure to preserve metals blanks.

11.3 A matrix spike, matrix spike duplicate and sample duplicate shall be performed for each waste type. At a minimum, follow the matrix spike addition guidance provided in each analytical method.

11.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP leaching of the sample.

11.5 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist.

11.6 Analyst Qualifications and Training

11.6.1 Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications for Chemistry* for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

12.1.1 Pace National's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

12.1.2 The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

12.1.3 All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions

were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace National's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

12.1.4 A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

12.1.5 Refer to laboratory SOPs ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

12.2.1 Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

13.0 Pollution Prevention And Waste Management

13.1 Pace National proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

13.2 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace National's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

14.1 NaOH solution is made up at 6N rather than 1N due to the large volume of Extraction Fluid #1 that is made up at a time.

14.2 Pressurizing the ZHE device during tumbling at 20psi instead of the method defined 5-10psi is performed due to the limitations of the pressure gauges on the extractors. The pressure at 30psi was found to not have a significant impact on the results of the analysis by the published method authors (Section 9.1.2, EPA Method 1311).

14.3 Samples that have a matrix that is incompatible with the ZHE apparatus (such as solvents which degrade the piston seals) may be tumbled in glass containers paying particular attention to keeping the headspace of the container to a minimum. These samples shall be clearly marked on the bench sheet with an explanation and also clearly qualified on the final test report.

14.4 Per specific client request, a variation of the solids determination in this procedure can be performed and reported. When TCLP_PPF is requested by a client, the percent of liquid that passes through the filter during the Sections 8.2 and 9.4 is reported to the client.

15.0 Responsibilities

15.1 Pace National employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace National's policy for temporary departure.

15.2 Pace National supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 Attachments

16.1 Not applicable to this SOP

17.0 References

17.1 Federal Register Part V Friday June 29, 1990 (Federal register/vol.51, No.216/Friday, November 7, 1986 Rules and Regulations Appendix 1 to Part 268--Toxicity Characteristic Leaching Procedure (TCLP)).

17.2 Toxicity Characteristic Leaching Procedure, SW-846 Method 1311, Revision 0, July 1992.

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: ENV-SOP—MTJL-0187 Toxicity Characteristic Leaching Procedure
TEST METHOD: EPA 1311

ISSUER: Pace National – Mt. Juliet, Tennessee

18.0 Revision History

Section	Description of Change	Date
9.2.2-9.2.23, revision history	Revised.	01/12/2022
All	Complete SOP reformat.	

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MTJL-0187	Toxicity Characteristic Leaching Procedure	03
ENV-SOP-MTJL-0187	Toxicity Characteristic Leaching Procedure	02
ENV-SOP-MTJL-0187	Toxicity Characteristic Leaching Procedure	01
ENV-SOP-MTJL-0187	Toxicity Characteristic Leaching Procedure	00
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	13
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	12
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	11
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	10
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	09
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	08
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	07
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	06
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	05
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	04
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	03
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	02
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	01
ESC Lab Sciences SOP #340358	Toxicity Characteristic Leaching Procedure	00

Pace Analytical®
ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MTJL-0215 **Revision:** 09

Document Title: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)

Department(s): Metals

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

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All dates and times are in Central Time Zone.

ENV-SOP-MTJL-0215

QM Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - Quality Program	22 Feb 2022, 08:21:41 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
[redacted]	Manager - EHS	02 Nov 2021, 09:11:02 AM	Approved
[redacted]	Manager - Operations	04 Nov 2021, 09:13:35 AM	Approved
[redacted]	Supervisor	04 Nov 2021, 12:04:12 PM	Approved

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)
ISSUER: Pace National – Mt. Juliet, Tennessee

1.0 SCOPE AND APPLICATION

STATE NOTE: For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0214.

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals and some non-metals in solution. This procedure follows the guidelines established in EPA method 200.7 and SW-846 Method 6010B, 6010C, and 6010D for drinking water, waste water, ground water, TCLP, SPLP, and STLC leachates, soils, sludge, sediments, solid wastes, oils, and other digestates after appropriate preparatory procedure is performed.

This procedure is also applicable to reporting calculated values for Calcium, Magnesium, and Total Hardness from values determined using EPA methods 200.7 or 6010B/C/D from groundwater, wastewater and drinking waters. Reporting limits for Hardness are derived from the annual MDL studies for Calcium and Magnesium of the appropriate determinative EPA method. The routine reporting limits for each category of hardness are listed in Table 1.2b.

1.2 This method is applicable for the analytes listed in Table 1.2a and b. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix, and instrument operating conditions. Table 1.2 also lists the Reporting Limits (RLs), used routinely by Pace Analytical National Center for Testing & Innovation (Pace National).

Table 1.2a: Environmental Analytes and Reporting Limits *(Subject to change, see section 13.1)*

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Aluminum	✓	✓	00.200	mg/L	✓	2.00	mg/Kg
Antimony	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Arsenic	✓	✓	00.010	mg/L	✓	1.00	mg/Kg
Barium	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Beryllium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Boron	✓	✓	0.050	mg/L	✓	5.0	mg/Kg
Cadmium	✓	✓	0.002	mg/L	✓	0.20	mg/Kg
Calcium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Chromium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Cobalt	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Copper	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Iron	✓	✓	0.100	mg/L	✓	10.0	mg/Kg
Lead	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Lithium	✓		0.015	mg/L	✓	1.50	mg/Kg
Magnesium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Manganese	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Molybdenum	✓	✓	0.005	mg/L	✓	0.50	mg/Kg
Nickel	✓	✓	0.010	mg/L	✓	1.00	mg/Kg

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STANDARD OPERATING PROCEDURE
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ISSUER: Pace National – Mt. Juliet, Tennessee

Analyte	Aqueous				Sediment		
	Ground Water/ Wastewater 6010B/C/D/200.7	Drinking Water 200.7*	RL	Units	Solids 6010B/C/D	RL	Units
Potassium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Selenium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Silicon	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Silver	✓	✓	0.005	mg/L	✓	5.00	mg/Kg
Sulfur	✓	✓	1.0	mg/L	✓		mg/Kg
Sodium	✓	✓	1.000	mg/L	✓	100	mg/Kg
Strontium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Thallium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Tin	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Titanium	✓	✓	0.050	mg/L	✓	5.00	mg/Kg
Vanadium	✓	✓	0.010	mg/L	✓	1.00	mg/Kg
Zinc	✓	✓	0.050	mg/L	✓	5.00	mg/Kg

**May not meet required Drinking Water Maximum Contamination Levels (MCLs) using this methodology.*

Table 1.2b: Hardness Categories and Reporting Limits

(Subject to change, see section 13.1)

Hardness:	RL (mg/L)
Calcium Hardness	1.25
Magnesium Hardness	0.41
Total Hardness	1.6

1.3 For the determination of total recoverable analytes in aqueous and solid samples, an acid digestion process is required. Environmental samples for analysis by Method 6010B, 6010C, or 6010D including, TCLP or EP leachates, soils, sludge, sediments, and other solid wastes require an acid digestion prior to analysis. Samples are digested by SW-846 methods 3005 (Acid Digestion of Waters for Total Recoverable Metals), 3010 (Acid Digestion of Aqueous Samples), 3015 (Microwave Digestion of Aqueous Samples), 3050 (Acid Digestion of Sediments, Sludge, Soil, and Oils) and 3051 (Microwave Assisted Digestion of Sediments, Sludge, Soil, and Oils). Digestion methods are found in ENV-SOP-MTJL-0217 and ENV-SOP-MTJL-0219.

1.4 The Clean Water Act has approved EPA Method 200.7 for demonstrating compliance on discharge monitoring for NPDES (National Pollution Discharge Elimination System) permits. 40 CFR136.3 has Guidelines for Establishing Test Procedures for Analysis of Pollutants. The National Primary Drinking Water Regulations for inorganic chemical sampling and analytical requirements can be found in 40 CFR141.23. Updates to these regulations can be found in the current Code of the Federal Register.

1.5 To determine dissolved analytes in aqueous samples, a 0.45µm filtration method is employed then the filtered samples are acidified. To reduce potential interferences, dissolved solids must be <0.2% (w/v).

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STANDARD OPERATING PROCEDURE
TITLE: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)
ISSUER: Pace National – Mt. Juliet, Tennessee

1.6 Analysis without acid digestion can be used for drinking water samples if the samples have been properly preserved with acid and have turbidity of <1 NTU at the time of analysis. These samples must be acidified to match the acid matrix of the calibration standards and analyzed directly. This total recoverable determination procedure is referred to as "direct analysis". Silver concentration cannot be determined from direct analysis when chloride ions are present as a silver chloride precipitate may be formed. The sample must be acid digested to form a soluble silver chloride complex. Some primary drinking water metal contaminants may require sample concentration to meet regulatory drinking water reporting limits criteria[14 2].

Method 6010D – Samples that are not digested necessitate the use of either an internal standard or should be matrix-matched with the standards. If using the former option, the instrument software should be programmed to correct for the intensity differences of the internal standard between samples and standards. **NOTE**: All samples analyzed by Method 6010 are typically digested.

1.7 When determining boron and silicon in aqueous samples, only plastic, PTFE (Teflon™) sample containers and laboratory glassware must be used. For accurate determination of boron in solid samples, only quartz or PTFE tubes must be used during acid digestion with immediate transfer of an aliquot of the final volume of digestate to a plastic centrifuge tube[14.2].

1.8 For the determination of titanium, white plastic and white printed containers must be avoided as titanium dioxide is used as a white pigment.

1.9 The total recoverable sample digestion procedure dissolves and maintains in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible following sample preparation[14.2].

1.10 Detection limits and linear ranges for the elements vary with the wavelength selected, the spectrometer, and the matrix. Table 1.11 provides a list of routinely used wavelengths and the type of spectrometer view used.

Method 6010D – IDLs are necessarily instrument-specific. Therefore, if needed, an IDL must be determined through a separate experimental study for each instrument. IDLs should be established, at a minimum, on an annual basis for each matrix and for each preparatory/determinative method combination used.

TABLE 1.10: WAVELENGTHS
(exact wavelengths vary slightly depending on the instrument)

Analyte	Wavelength (nm)	Type of View
Aluminum	308.215	Radial
Antimony	206.836	Axial
Arsenic	188.979	Axial
Barium	233.527	Axial
Beryllium	313.107	Radial
Boron	249.772	Radial
Cadmium	214.440	Axial
Calcium	317.933	Radial

Analyte	Wavelength (nm)	Type of View
	373.690	
Chromium	205.560	Axial
Cobalt	228.616	Axial
Copper	324.752	Radial
Iron	259.940 271.441	Radial
Lead	220.353	Axial
Lithium	670.784	Radial
Magnesium	279.077	Radial
Manganese	257.610	Axial
Molybdenum	202.031	Axial
Nickel	232.003	Axial
Potassium	766.490	Radial
Phosphorus	177.495	Axial
Selenium	196.026	Axial
Silicon	251.611	Axial
Silver	328.068	Axial
Sodium	589.592 818.326	Radial
Strontium	407.771	Radial
Sulfur	181.972	Axial
Thallium	190.801	Axial
Tin	189.927	Axial
Titanium	334.940	Radial
Vanadium	292.402	Radial
Zinc	213.857	Axial

1.11 Users of the data generated using this method must state the data-quality objectives (DQOs) prior to analysis.

1.12 Any deviations from this SOP must be documented. Deviations are reflected in a case narrative and the method is reported as modified. Per customer requirement, the procedure and QC criteria described in this SOP can be changed/modified. Authorization from the Operations Manager and Project Manager is required for each modification and Regulatory Affairs approval must also be secured for any deviation.

1.13 An MDL study must be completed at least annually or more frequently if major instrumentation changes occur. Method Detection Limits (MDLs) are performed based on ENV-SOP-MTJL-0016. Updated MDL records are filed and stored in a central location within the department.

1.13.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ) studies are completed at the frequency required by the TNI standard per the procedure identified in the ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD), and Limits of Quantitation (LOQ).* Should the procedure be utilized for DoD support; then the frequency of these studies must meet the requirements of the current DoD QSM.

1.14 Linear Dynamic Range (LDR) and Inter-element correction factor (IEC) studies must be analyzed semi-annually for each analytical instrument or when there are major changes/repairs to the instrument[14.5, 14.1]. Instrument Detection Limit studies must be analyzed at least quarterly for each analytical instrument[14.5].

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are aspirated into the nebulizer and the resulting aerosol is transported to the plasma torch. The emission spectra are dispersed by a grating spectrometer separating the light emitted into the distinct wavelengths generated by each element in the sample. A photosensitive device monitors the intensities of each wavelength line in the spectra. The intensity of light on the photosensitive device produces a signal that is measured and processed by a computer system. Due to the many possible wavelengths of light generated by each element and possible overlapping of high intensity peaks, a background correction technique is required for trace element determination. Background intensities must be measured adjacent to the analyte spectra lines during analysis. The position selected for background intensity measurement can be selected on either or both sides of the analyte wavelength line and must be determined by the complexity of the spectrum adjacent to the analyte line. The position used for background correction must be as free from spectral interference as possible and must reflect the same change in background intensity as occurs at the analyte wavelength. Background correction is not required in cases of line broadening where the background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

2.2 Dissolved Analyte - The concentration of analyte in an aqueous sample that has been passed through a 0.45µm membrane filter assembly prior to sample acidification and digestion.

2.3 Total (Total Recoverable) Analyte – The concentration of analyte determined either by ‘‘direct analysis’’ of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method

2.4 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength. The IDL assures with 99% certainty that a value is above the instrument noise level.

Note: An IDL is a statistical determination without analytes present used to assess background correction protocols and an MDL is determined with low levels of analytes present to determine instrument sensitivity for each analyte.

2.5 Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration remains linear.

2.6 Plasma Solution - A solution that is used to determine the optimum torch height relative to the radio frequency (RF) coil for viewing the spectrum.

2.7 Interference Check Sample (ICS) – A series of five solutions (ICSA, ICSAB, ICSA2, LA, & CE) to verify that inter-element interferences are correctly compensated. The ICS checks provide an adequate on-going test of inter-element correction (IEC) factors. These standards are referred to the Spectra Interference Check (SIC) in EPA Method 200.7

2.7.1 ICSA – A solution containing only the interfering analytes at high concentrations.

2.7.2 ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.

2.7.3 ICSA2 – A solution containing interfering analytes not contained in the ICSA.

2.7.4 LA – A solution containing Lanthinum at a high concentration.

2.7.5 CE – A solution containing Cerium at a high concentration.

2.8 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking water, surface water, ground water, storm water, industrial or domestic wastewater.

2.9 Preparation Batch - For method 6010B/C/D/ EPA 200.7 (WW only): A group of samples (not to exceed twenty) of a similar matrix, which have been digested at the same time using the same digestion process and have all necessary QC associated with them. For method 200.7 (DW only): A group of samples (not to exceed ten) of similar matrix, which have been digested at the same time using the same digestion process and have all necessary associated QC.

2.10 Analytical batch - A group of samples that are analyzed in the same sequence with all appropriate preparation and analytical QC.

2.11 Inter-element correction (IEC) coefficient - analyte concentration equivalent arising from a given interferent's concentration.

2.12 Serial Dilution - a dilution and reanalysis of a field sample that is performed once per batch of samples. One sample is diluted 5X and reanalyzed.

2.13 Post Spike – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch.

2.14 Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D; the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.

2.15 See the current Quality Assurance Manual for other definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Samples submitted for analysis that do not meet the requirements contained within this section must be addressed before performing the logging process within the laboratory. In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the

client approves the completion of the analytical process, sample results can be qualified per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

4.3 Prior to the collection of an aqueous sample, consideration must be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pre-treatment steps can be taken. The pH of all aqueous samples must be assessed immediately prior to sample digestion or "direct analysis" to ensure the sample has been properly preserved. If the field sample is properly preserved, the sample can be held up to 6 months prior to analysis.

4.4 For the determination of dissolved elements, the sample must be filtered through a 0.45µm pore diameter membrane filter to remove the suspended elements or particles. This filtration must take place at the time of collection or as soon thereafter as practically possible. Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus must be used when the determinations of boron and silica are critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH <2.

4.5 For the determination of total recoverable elements in aqueous samples, samples must not be filtered, but acidified with (1:1) nitric acid: water to pH <2. Preservation may be done at the time of collection; however, to avoid the hazards of strong acid use in the field, possible transport restrictions, or possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample must be mixed and equilibrated for 24 hours. The pH is verified at <2 prior to withdrawing an aliquot for acid digestion or "direct analysis". If, for reasons such as high alkalinity, the sample pH is verified to be >2, more acid must be added and the sample equilibrated for another sixteen hours until verified to be pH <2.

4.6 Solid samples require no preservation prior to analysis. Solid samples can be held up to six months from the time of sample collection until preparation and analysis.

4.7 For aqueous samples, a field blank must be prepared and analyzed as required by the data user. Use the same container and preservative as is used in field sample collection. The sample holding time is six (6) months from the date and time of collection until analysis. Samples are preserved to pH <2 with nitric acid.

5.0 INTERFERENCES

5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

5.1.1 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. The location(s) selected for the measurement of background intensity is determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. Changes in background correction must be saved in the instrument

method. Background correction can be established by scanning the following three solutions: 1) blank (same as calibration blank); 2) solution, containing analytes at significant concentration to raise a signal above background signal (CCV solution may be used) at mid-range of the curve; 3) solution(s) containing most common interfering elements at high concentration and other interferents as well (ICSAB solution may be used).

5.1.2 Spectral overlaps can be compensated for by equations that correct for inter-element contributions, which involve measuring the interfering elements. When operative and uncorrected, these interferences produce false-positive determinations and are reported as analyte concentrations. Users may apply inter-element correction factors determined on their instruments within tested concentration ranges to compensate (offline or online) for the effects of interfering elements. Consult the method for specific identified interferences.

5.1.3 When inter-element corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions. The IEC's are established by analyzing a solution of the interfering element at a high concentration within the LDR limit, measuring the analyte concentration equivalents arising from the interfering element, calculating the interference factor as analyte reading in mg/L, then dividing by the interfering element concentration. The IEC's are changed in the stored ICP instrument method. Inter-element corrections vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples might contain uncommon elements that could contribute spectral interferences.

5.1.4 Interference effects must be evaluated for each individual instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). To determine the appropriate location for offline background correction, the user must scan the area on either side of the peak adjacent to the wavelength and record the apparent emission intensity from all other method analytes. The location selected for background correction must be either free from offline inter-element spectral interference or a computer routine must be used for their automatic correction on all determinations.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as using a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This can be controlled using a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that

better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision. This is accomplished with the use of mass flow controllers.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching and by standards addition procedures. Chemical interferences are highly dependent on matrix type.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them.

5.5 Linear Dynamic Range (LDR) study is performed by analyzing a solution of each element at maximal concentration unless the result falls outside 10% RPD. The highest calibration standard for each analyte cannot be greater than the LDR for that analyte. If an interferent is found greater than the LDR and an IEC factor is established between the interferent and analyte of interest, the sample must be diluted for proper correction of inter-element interferences. Instrument methods with different calibration standard concentrations require separate LDR studies.

5.6 Background correction is performed as needed and LDR and IEC studies are completed as required by each published analytical method and whenever significant changes to instrumentation are made. Background, or blank matrix, subtraction is not performed for environmental samples.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively coupled plasma emission spectrometer:

6.1.1 Perkin Elmer Model 5300 or Thermo Model 7000 series ICP, or equivalent, with background correction and computer control

6.1.2 Cetac Autosampler or ESI autosampler

6.1.3 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.

6.2 Narrow-mouth storage bottles, FEP (fluorinated ethylene propylene) with screw closure, 125mL to 1L capacities

6.3 One-piece stem FEP wash bottle with screw closure, 125mL capacity

6.4 Adjustable pipettes (Eppendorf or equivalent), ranges from 2µL to 5000µL

6.5 Class A volumetric flasks for standards preparations

6.6 Polypropylene (PP) conical tubes

6.7 Peristaltic pump

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected unless otherwise noted.

7.2 Hydrochloric acid, concentrated (sp. gr. 1.19) - HCl

7.2.1 Hydrochloric acid (1+1) - Add 500mL concentrated HCl to 400mL reagent water and dilute to 1L with reagent water.

7.3 Nitric acid, concentrated (sp. gr. 1.41) - HNO_3

7.3.1 Nitric acid (1+1) - Add 500mL concentrated HNO_3 to 400mL reagent water and dilute to 1L with reagent water.

7.4 Laboratory Reagent water

7.5 ICP Standard List

Calibration	Stock Std Cat #	Working Standard Preparation
RL Standard #1	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
0.5ppm Standard #2	HP6928-1L	125mL of 2.0ppm Std, 10% rinse to volume 500mL
1ppm Standard #3		250mL of 2.0ppm, 10% rinse to volume 500mL
2ppm Standard #4		Direct pour
10ppm Standard #5	HP6409-500	Direct pour
250ppm Standard #6	HP4526-1L	500mL of 500ppm Std, 10% rinse to volume 1000mL
500ppm Standard #7		Direct pour
La Standard #8	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL

Standards	Stock Std Cat #	Working Standard Preparation
ICV	ESC-8	5mL of Stock ICV Std, 10% rinse to volume 500mL
CCV	HP6929-1L	50mL of Stock CCV Std, 10% rinse to volume 1000mL
ICVLL	HP7013-500	50mL of Stock LL Std, 10% rinse to volume 1000mL
CCVLL		50mL of Stock LL Std, 10% rinse to volume 1000mL

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STANDARD OPERATING PROCEDURE
TITLE: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)
ISSUER: Pace National – Mt. Juliet, Tennessee

Standards	Stock Std Cat #	Working Standard Preparation
ICSA	ICL500-6	50mL of Stock A Std, 10% rinse to volume 500mL
ICSAB	ICL500-6,HP2739-500	50mL of Stock A Std, 5ml of Stock AB Std, 10% rinse to volume 500mL
CE	HP100010-1	5mL of Stock Ce Std, 10% rinse to volume 500mL
ICSA2	varies	5 mL of Stock Std, 10% rinse to volume 500mL
LA	VHG-PLAN-100	10mL of Stock La Std, 10% rinse to volume 1000mL.
IEC / LDR As	HP10003	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Co	HP100013	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cr	HP100012	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Cu	HP100014	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Mn	HP100032	10mL of Stock As Std, 10% rinse to volume 1000mL
IEC / LDR Si	HP100050	10mL of Stock As Std, 10% rinse to volume 1000mL
Internal Std	HP10M67-1,HP10M24-1	1mL of Stock Y, 3mL of Stock In, 10% rinse to volume 1000mL
10% Blank and Rinse	A200C-212	Fill container to 90% with DI water, add HNO3 to volume

7.6 Blanks - Three types of blanks are required for ICP-AES analysis

7.6.1 The calibration blank is used to establish the baseline for the instrument prior to the analysis of the analytical curve. The calibration blank is prepared by acidifying reagent water to the same acid concentration as used for the standards (10% HNO3).

NOTE: The calibration blank must be stored in a FEP bottle to minimize leaching from other container materials that can cause an elevation in the target analytes leached causing an inherent bias in the calibration and quantitation of field samples when baselines are established prior to calibration of the ICP-AES.

7.6.1.1 Following calibration, the Initial Calibration Blank (ICB) is analyzed prior to field sample analyses. A Continuing Calibration Blank (CCB) is analyzed following the CCV after every ten samples and at the end of the analytical sequence to verify on-going acceptable instrument conditions.

7.6.2 The method blank is used to assess possible contamination from the sample preparation procedure. The method blank must contain all the reagents in the same volumes as used in sample preparation. The method blank must be

prepared in the same manner as the samples including sample digestion, when applicable.

7.6.3 The rinse blank is prepared by acidifying reagent water to the same concentrations as the acids as used in the calibration blank (10% HNO3). This solution is stored in a convenient manner. The rinse blank is used for equipment "wash out" to flush the sample delivery system and eliminate memory effects (carryover) from previous samples or standards.

7.7 Mixed Calibration Standard Solutions are used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. See section 7.5 above for preparation of these standards.

Concentration of Target Analytes in Calibration Standards in mg/L

Analyte	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Silver	0.005	0.5	1.0	2.0				
Aluminum	0.2				10	250	500	
Arsenic	0.01	0.5	1.0	2.0				
Boron	0.2		1.0	2.0				
Barium	0.005	0.5	1.0	2.0	10			
Beryllium	0.002	0.5	1.0	2.0				
Calcium	1.0		1.0	2.0	10	250	500	
Cadmium	0.002	0.5	1.0	2.0				
Cerium						10		
Cobalt	0.01	0.5	1.0	2.0				
Chromium	0.01	0.5	1.0	2.0				
Copper	0.01	0.5	1.0	2.0				
Iron	0.10	0.5	1.0	2.0	10	100	200	
Potassium	1.0			2.0	10	50	100	
Phosphorus	0.1	0.5	1.0	2.0				
Lanthanum								10
Lithium	0.015	0.5	1.0					
Magnesium	1.0				10	250	500	
Manganese	0.01	0.5	1.0	2.0				
Molybdenum	0.005	0.5	1.0	2.0				
Sodium	1.0		1.0	2.0	10	250	500	
Nickel	0.01	0.5	1.0	2.0				
Lead	0.005	0.5	1.0	2.0				
Antimony	0.01	0.5	1.0	2.0				
Selenium	0.01	0.5	1.0	2.0				
Silicon	0.2	0.5	1.0	2.0	10			
Strontium	0.01	0.5	1.0	2.0				
Sulfur	1.0		1.0	2.0	10	50	100	
Tin	0.05	0.5	1.0	2.0				
Thallium	0.01	0.5	1.0	2.0				
Vanadium	0.02	0.5	1.0	2.0				
Zinc	0.05	0.5	1.0	2.0				
Titanium	0.05	0.5	1.0	2.0				

7.8 Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level ICV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	1.0
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	1.0
Beryllium	1.0
Calcium	10.0
Cadmium	1.0
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	10.0
Lithium	1.0
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	1.0
Sodium	10.0
Nickel	1.0
Lead	1.0
Antimony	1.0
Selenium	1.0
Silicon	1.0
Strontium	0.4
Tin	1.0
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0
Sulfur	10.0

7.9 Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. EPA Method 200.7 refers to this standard as the Instrument Performance Check (IPC) standard. See Section 7.5 above for the preparation of this standard.

All analytes are present in the mid-level CCV solution at the following concentrations (mg/L):

Analyte	Concentration
Silver	0.5
Aluminum	10.0
Arsenic	1.0
Boron	1.0
Barium	0.50
Beryllium	0.20
Calcium	50.0
Cadmium	0.50
Cobalt	1.0
Chromium	1.0
Copper	1.0
Iron	10.0
Potassium	50.0
Lithium	1.0

Analyte	Concentration
Manganese	1.0
Molybdenum	0.25
Sodium	50.0
Nickel	1.0
Lead	0.50
Antimony	0.5
Selenium	1.0
Silicon	2.0
Strontium	1.0
Tin	0.5
Thallium	1.0
Vanadium	1.0
Zinc	1.0
Titanium	1.0

Analyte	Concentration
Magnesium	10.0
Phosphorus	1.0

Analyte	Concentration
Sulfur	5.0

7.10 Low Level Initial/Continuing Calibration Verification for EPA 6010C (ICVLL/CCVLL) –The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards. See Section 7.5 above for the preparation of this standard.

The concentration of the low-level ICVLL/CCVLL solution is listed in the table below:

Analyte	Concentration (mg/L)
Silver	0.005
Aluminum	0.2
Arsenic	0.01
Boron	0.2
Barium	0.005
Beryllium	0.002
Calcium	1
Cadmium	0.002
Cobalt	0.01
Chromium	0.01
Copper	0.01
Iron	0.10
Potassium	1
Lithium	0.015
Magnesium	1
Phosphorus	0.1

Analyte	Concentration (mg/L)
Manganese	0.01
Molybdenum	0.005
Sodium	1
Nickel	0.01
Lead	0.005
Antimony	0.01
Selenium	0.01
Silicon	0.2
Strontium	0.01
Tin	0.05
Thallium	0.01
Vanadium	0.02
Zinc	0.05
Titanium	0.05
Sulfur	1.00

7.11 Interference Check Solutions (ICSA, ICSAB, ICSA2, LA, CE) – The ICS checks are prepared to contain known concentrations of interfering elements that provides a test of the correction factors. The ICSA, ICSA2, LA, and CE solutions contains the interfering elements at a high concentration and the ICSAB contains both the interfering analytes at a high concentration and the analytes of interest at 0.5 to 1.0mg/L. EPA Method 200.7 and 6010D refers to this standard as the Spectral Interference Check (SIC) standard. See Section 7.5 above for the preparation of these standards.

7.11.1 The ICSA solution contains 5000mg/L of each Al, Ca, Mg and 2000mg/L Fe.

7.11.2 The ICSAB solution contains all the components at the same concentrations of the ICSA and other target analytes of interest spiked. In the working ICSAB solution, silver, boron, cadmium, nickel, lead, silica, and zinc are present at 1.0mg/L. All other analytes (arsenic, barium, beryllium, cobalt, chromium, copper, manganese, molybdenum, antimony, selenium, tin, thallium, vanadium and titanium) are present at 0.5mg/L.

7.11.3 The ICSA2 solution contains interfering elements that are not in the ICSA. In the working ICSA2 solution, all analytes are present at 10mg/L.

7.11.4 The LA solution contains 10mg/L of La.

7.11.5 The CE solution contains 10mg/L of Ce.

7.12 The IEC / LDR solutions are prepared to contain known concentrations of interfering elements that are used to adjust the IECs and are used for daily LDRs for 6010D. The interfering elements are present at 10mg/L.

7.13 Internal Standards – The internal standard response is used to measure the relative responses of other method analytes in each sample. Yttrium and Indium are used as internal standards. See Section 7.5 above for the preparation of this standard.

7.14 For the preparation of Laboratory Control Samples (LCSs) and Matrix Spikes (MSs) see the applicable sample preparation SOPs.

8.0 PROCEDURE

8.1 Sample Analysis

8.1.1 **Initializing the Instrument:** Prior to daily calibration of the instrument, inspect the sample introduction system including the nebulizer, torch, injector tube for salt deposits, dirt, and debris that would restrict solution flow and affect instrument performance.

8.1.1.1 Replace the uptake tubing daily.

8.1.1.2 If any of the sample introduction parts appear soiled, first remove the part from the instrument by following the maintenance procedure in the instrument manual. Once removed, attempt to clean the part with a dilute solution of 5% nitric acid. Cleaning may be performed using a cotton swab or by submersing the part in the acid solution for no longer than five (5) minutes. If cleaning is successful, dry the part using compressed air or argon and replace it in the instrument. If cleaning does not adequately remove the residue, the part must be replaced with a new one in accordance with the manufacturer's directions. Replacement parts are kept in the cabinet in the instrument lab.

8.1.2 **Instrument Stability:** The instrument must be allowed to become thermally stable before calibration and analyses. This usually requires at least 30 minutes of operation.

8.1.3 **Instrument Calibration:** For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. A peristaltic pump is used to introduce all solutions, samples, and the internal standard to the nebulizer. To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before obtaining the sample analyte response.

8.1.3.1 Use the average value from three replicate analyte responses per sample to be correlated to the overall analyte concentration in the solution being sampled. Flush the system with the rinse blank for a minimum of 60 seconds between each standard.

8.1.3.2 The calibration regression is generated using first order linear regression of a calibration blank and three calibration standards where each element is present. The blank is included as a point in the calibration curve to determine the baseline correction needed for the instrument to effectively quantitate target analyte concentrations.

8.1.3.3 Calibration acceptance criteria are described in section 10.4.

8.1.4 **Internal Standard:** All standards/samples/QC etc. contain yttrium and indium as the internal standards. The instrument adds the internal standards automatically. The instrument injects a constant volume into each solution being analyzed (i.e. standard, blank, field sample, LCS/LCSD/MS/MSD/DUP) and monitors the intensity at the sample level. An internal standard is the chosen alternative to the method of standard additions (MSA). If signal variation results from the sample introduction system (samples of different viscosity, matrix constitution), all the elements are corrected in the same way by an internal standard. If variation results from a variation of the energy transfer, the internal standard most accurately corrects elements of similar energy. Internal standard acceptance criteria are described in section 10.14.

8.1.5 **Calibration Accuracy:** Verify the acceptable initial calibration of the instrument using a standard source that is either an independent lot or entirely different manufacturer to ensure calibration accuracy. After calibrating and rinsing the instrument, analyze the ICV and, if analyzing samples using EPA 6010C, analyze the ICVLL standards. These standards are prepared as directed in section 7.7 and 7.9. Acceptance criteria are described in section 10.5.

8.1.6 **On-going Calibration Stability:** Verify the acceptable on-going instrument calibration by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples using the CCV and CCB and at the end of the analytical run using the CCV, CCVLL (if analyzing EPA 6010C samples), and CCB that must meet the criteria described in sections 10.6 & 10.14.

8.1.7 **Accurate Background Corrections:** The interference check standards (ICSA, ICSAB, ICSA2, LA, and CE) are used to verify the inter-element and background correction factors at the beginning of an analytical run. The ICSA and ICSAB are verified during every 8-hour work shift. The interference check standards must meet the criteria found in section 10.10.

8.1.8 An Initial Calibration Blank (Section 7.5.1) is analyzed before sample analysis is initiated to verify the cleanliness of the analytical system. Acceptance criteria are described in section 10.14.

8.1.9 **Field Sample Analysis:** After completion of the above calibration requirements, samples must be analyzed in the same operational manner used in the calibration routine with the rinse blank also being used between all sample solutions, method blanks, Laboratory Control Standards, matrix spike, matrix spike duplicates, and check solutions.

8.1.10 **Dilutions:** If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte's determined Linear Dynamic Range (LDR), see section 10.4.2 for further guidance.

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STANDARD OPERATING PROCEDURE
TITLE: Determination Metals and Trace Elements in Various Matrices by ICP-AES (EPA Methods 6010B, 6010C, 6010D [ICP-OES], and 200.7) Including Hardness (EPA Methods 200.7 and 6010B/C/D and SM 2340B)
ISSUER: Pace National – Mt. Juliet, Tennessee

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/kg dry weight corrected for solid samples.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with compensation for sample dilution. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentrations.

9.3 For total recoverable aqueous analytes, multiply solution analyte concentrations by the dilution factor 0.5 when a 100mL aliquot is used to produce the 50mL final digestate volume, and report data. If a different aliquot volume other than 100mL is used for sample preparation, adjust the dilution factor accordingly. Account for any additional dilution of the prepared sample digestate required to complete the determination of any analytes exceeding 90% or greater of the LDR upper limit. Never report analyte concentrations below the MDL and if reporting between the MDL and the routine RL, results should be qualified with the appropriate indicator for estimated target analyte concentration. Routine reporting limits are adjusted for any dilution required by the sample analysis.

9.4 Results are reported to Three significant figures by the laboratory LIMS. Analyte concentrations for solids data should be rounded in a similar manner following dry weight corrections.

9.5 For total recoverable analytes in solid samples, calculate the target analyte concentration using the equation below and do not report analyte data below the estimated solids RL or an adjusted RL based on additional dilutions required to complete the analysis:

$$\text{Sample Conc. (mg/Kg) dry-weight basis} = \frac{C \times V \times D}{W}$$

where: C = Concentration in extract (mg/L)
V = Volume of extract (L, 100mL = 0. 1L)
D = Dilution factor (undiluted = 1)
W = Weight in Kg of sample aliquot extracted (g x 0.001 = Kg)

9.6 Soil samples are routinely reported on a dry weight basis. Soil samples must be processed using the ENV-SOP-MTJL-0065, *Total Solids*. After a dry weight for each sample has been obtained, the calculations are performed automatically by the laboratory LIMS as follows:

$$\%\ \text{solids (S)} = \frac{DW}{WW} \times 100$$

where: DW = Sample weight (g) dried
WW = Sample weight (g) before drying

9.7 Hardness calculations:

Total Hardness, mg equivalent $CaC0_3$/L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]
Calcium Hardness = 2.497 [Ca, mg/L]
Magnesium Hardness = 4.118 [Mg, mg/L]

9.8 To calculate the silica concentration from silicon analysis:

Silica (mg/L) = 2.14*[Silicon, mg/L]

9.9 Formula needed to calculate dilution of stock standards of known concentration to a known final volume, using the basic chemistry formula, $C_1*V_1=C_2*V_2$:

$$V_{stock} = V_{std} * C_{std} / C_{stock}$$

where: V_{stock} = volume of stock standard required (mL)
V_{std} = final volume of diluted standard required (mL)
C_{stock} = concentration of stock standard required (µg/mL or µg/L)
C_{std} = final concentration of diluted standard required (µg/mL or µg/L)

NOTE: Be sure to maintain consistent units for both concentration and volume during the use of the calculation and keep in mind that (1µg/mL = 1mg/L=1000µg/L, 1L=1000mL=1000000µL, and 1mL=1000µL)

9.10 Percent Relative Intensity (%RI) for internal standard assessment (ISTD):

%RI = Intensity of $ISTD_{sample}$ / Intensity of $ISTD_{CalBlk}$ * 100%

9.11 Relative Standard Error (RSE – expressed as a percentage)

$$RSE \text{) } 100 \text{ (} \sqrt{\sum_{i)1}^{n} \left[\frac{x'_i \text{ ! } x_i}{x_i} \right]^2 \Big/ (n \text{ ! } p)}$$

where:
x'_i = Measured amount of analyte at the calibration level *i*, in mass or concentration units
x_i = True amount of analyte at calibration level *i*, in mass or concentration units
p = Number of terms in the fitting equation (average – 1, linear = 2, quadratic = 3)

9.12 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications,* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.1.1 Prior to using Method 6010D for quantitation of samples, an initial demonstration of performance packet must be completed. This packet must document:

+ The selection criteria for background correction points
+ Analytical dynamic ranges including the applicable equations and upper limits of ranges

+ IDLs and Method LLOQs
+ The determination and verification of interelement correction equations or other routines for correcting spectral interferences. These data must be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. The data must be kept on file and available for review by the data user or auditor.

10.2 Use Prep Data to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Batch Analyses

10.3.1 Environmental Preparation Batches: Preparation batches are defined as sets of 1-20 samples as defined in Chapter 1 of SW-846 and in section 9.3.1 of EPA 200.7. Preparation batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Laboratory Control Sample Duplicate (LCSD), 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Matrix Spike/Spike Duplicate (MS/MSD) pair. All batch information is maintained in Prep Data computer program.

10.3.2 Analytical Batches: Analytical batches are defined as a sequence of samples analyzed concurrently using the same calibrated instrument. Analytical batches include the QC samples produced in the Preparation Batches, in addition to: 1 Serial Dilution, 1 Sample Post Digestion Spike, 1 Initial Calibration Verification (ICV) following initial calibration, 1 Initial Calibration Verification-Low Level (ICVLL) following initial calibration (when analyzing EPA 6010C, 6010D and DOD only) 1 Initial Calibration Blank following the ICVLL, 1 Continuing Calibration Verification (CCV) following each 10 samples and at the conclusion of the sequence, 1 Continuing Calibration Verification-Low Level (CCVLL) at the conclusion of the sequence (when analyzing EPA 6010C only), 1 Continuing Calibration Blank (CCB) following each CCV, 1 Interference Check Sample A (ICSA), 1 Interference Check Sample AB (ICSAB), Interference Check Sample 2, 1 Lanthinum Interference Check Sample, and 1 Cerium Interference Check Sample following each initial calibration. A (ICSA) and 1 Interference Check Sample AB (ICSAB) at the end of the sequence or at least twice per each eight (8) hour shift. All batch information is maintained in Prep Data computer program.

10.4 Supporting Analytical Studies

10.4.1 Instrument Detection Limits (IDL) Studies - IDLs in µg/L can be determined as the mean of the calibration blank results plus three times the standard deviation of 10 replicate analyses of the solution. Use zero for the mean if the mean is determined to be a negative value.

IDLs must be verified quarterly[14.13] or when major instrumentation change occurs.

10.4.2 Linear Dynamic Range (LDR) Studies – Linear dynamic ranges are established for each instrument to allow for quantitation above the highest level of calibration without qualification. ICP instruments are known to remain linear at high levels, but each upper limit of linearity is based on the target analyte being measured and the routine instrument operating conditions.

To perform a linear dynamic range study, the instrument must be calibrated normally as used with client field samples. The LDR is determined by the

analysis of a minimum of three, but preferably five, different increasing concentrations of standards containing each target analyte across a range. One concentration should be near the expected upper linear range for each analyte. The highest concentration, where the instrument calibration remains linear, is determined when the observed concentration of the increasing standards is no more than 10% below the expected concentration of the analyte. If more than a 10% deviation exists, the instrument is proven to no longer be linear at that value for that analyte. The upper linear range is therefore the next lower concentration of standards used in the determination. Samples quantitated above that upper determined LDR require dilution to quantitate within the proven linear range of the instrument.

LDR studies must be verified semi-annually[14.1] or when major instrumentation change occurs.

Method 6010D - LDR standards must be ran daily within ten percent of true value, or dilute all samples above the high standard in the curve.

STATE NOTE: For work performed in support of the NC Department of Natural Resources (15A NCAC 02H.0805(a)(7)(l)) for target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

10.4.3 Method Detection Limits – See also ENV-SOP-MTJL-0016, *Method Detection Limits (MDL), Limits of Detection (LOD) and Limits of Quantitation (LOQ)*.

MDL studies are required annually or when instrumentation change occurs. Method detection limit studies are performed on blank matrices most closely matching field sample matrices.

10.4.4 Inter-element Correction Factors – All inter-element spectral correction factors must be verified and updated every six months or when major instrumentation change occurs.[14.1,14 5,].

Criteria for determining an inter-element spectral interference is an apparent positive or negative concentration of an analyte that is outside the 3-sigma control limits of the calibration blank for the analyte. See Attachment II for a listing of potential interfering analytes and their contributions from SW-846 method EPA 6010B. Testing is performed using 100mg/L single element solutions; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

Suggested analytes that are known to commonly interfere include: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V, and Zn.

10.4.5 Proficiency Testing (PT) – See also ENV-SOP-MTJL-0022, *Proficiency Testing Program*. Proficiency testing is performed in the metals department in support of both environmental and industrial hygiene analyses. Environmental PTs are performed semi-annually for Water Supply (Safe Drinking Water Act), Water Pollution (Clean Water Act), and soils (RCRA) testing.

10.5 Initial Calibration - Run a calibration curve on a daily basis that employs a minimum of a calibration blank and three standards for each target analyte. If the correlation coefficient does not meet the acceptance criteria, see the corrective action guidance listed in Section 11.1.

NOTE: For EPA Methods 200.7 & 6010B/D, the linear regression correlation coefficient for the each analyte in the calibration curve lines must be ≥0.995.

NOTE: For Method 6010D - If a multipoint calibration is used the low standard must be at or below the LLOQ.

NOTE: For EPA Method 6010C, the regression correlation coefficient must be ≥0.998.

10.6 Initial Calibration Verification (ICV/ICVLL) - Verify the accuracy of the initial instrument standardization by analyzing appropriate check standards following calibration. The routine mid-level ICV must be prepared from a source that is independent of the stock standard used for the preparation of the initial calibration curve. For EPA Method 6010C, a low-level ICV (ICVLL) is also performed as required; however the low level ICV is not required to be from a second source. It may be made from the same stock standard as the calibration standards as long as the initial calibration is verified by a second source in the mid-level ICV.

10.6.1 **EPA Method 6010B/D** - The routine ICV standard recovery results must be ±10% of the true value for EPA method 6010B/D. The RSD must be <5% for the triplicate passes of the spectrometer. If the RSD exceeds 5% and/or the recovery exceeds 10%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.2 **EPA Method 200.7** - The routine ICV standard recovery results must be ± 5% of the true value for EPA method 200.7. The RSD must be within 3% for the four replicate passes of the spectrometer. If the RSD exceeds 3% and/or the recovery exceeds 5%, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.3 **EPA Method 6010C -** The routine ICV standard recovery results must be ±10% of the true value for EPA methods 6010C. The RSD must be <5% for the triplicate passes of the spectrometer. The ICVLL standard recovery results should be within 70-130%. If the recovery does not meet the criteria for either level of ICV and/or the %RSD is exceeded, locate and correct the cause of the problem and do not proceed until this criterion is met. Corrective actions for failures can be found in section 11.2.

10.6.4 **EPA Method 6010D** Low-level Readback or Verification - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.

10.6.5 **EPA Method 6010D** Mid-level Readback or Verification - For a multi-point calibration, the midlevel standard should quantitate to within 90-110% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the midpoint of the linear range is analyzed and should recover within 90-110% of the true value.

10.7 Continuing Calibration Verification (CCV/CCVLL) - Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Verification is achieved by analyzing both a CCV standard and a CCB (instrument blank). Continuing calibration verification standards can be created from either primary or secondary source standards from those used in instrument calibration. Continuing instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria per the method being analyzed:

10.7.1 **For SW-846 Method 6010B/D** – Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within ±10%. The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within ±50%.

10.7.2 **For EPA Method 200.7 -** Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within ±10%. The RSD must be within 3% for the triplicate passes of the spectrometer.

10.7.3 **For SW-846 Method 6010C** - Continuing calibration verification (CCV) analyzed after every 10 samples and at the conclusion of the sequence must have a recovery within ±10%. The RSD must be within 5% for the triplicate passes of the spectrometer. The CCVLL recovery should be within ±30%. ICVLL are analyzed at the beginning of the analytical run and CCVLL are analyzed at the end of the analytical run for 6010C.

10.8 Method/Calibration/Rinse Blanks

10.8.1 Method Blank

10.8.1.1 A method blank is generated for each analytical batch during sample preparation to determine if any contamination is introduced during sample processing. A method blank is routinely a volume of reagent water that is carried through the entire digestion and analysis procedure with the samples.

10.8.1.2 The method blank must not contain analytes >MDL or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium. If target analytes are present in the method blank, corrective action must be taken. See section 11.5 for corrective actions.

NOTE: Per DoD QSM, version 5.0, Section 1.7.4.1, DoD/DOE require that method blanks be evaluated to ½ RL (LOQ) for target analytes and RL (LOQ) for common laboratory contaminants. If contaminants are present in the blank above this level,

samples must be re-prepared and re-analyzed or reported with appropriate qualification.

NOTE: Method 6010D – The method blank is considered to be acceptable if target analyte concentrations are less than ½ the LLOQ or are less than project-specific requirements.

State Note: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.

State Note: For West Virginia samples analyzed by Method 6010D, blanks are generally considered to be acceptable if target analyte concentrations are less than ½ the lower limit of quantitation (LLOQ) or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project.
For West Virginia samples analyzed by Method 200.7, blank values that exceed the MDL indicate laboratory or reagent contamination should be suspected.

10.8.2 Calibration Blank

10.8.2.1 An initial calibration blank is generated for each analytical sequence using acidified reagent water. The CALBLK analyzed prior to the initial calibration standards is used to establish a baseline for the instrument prior to calibration. Great care is required during this analysis to ensure that the baseline is correctly established prior to the calibration of the instrument and the analysis of field samples. Inaccurate baselines established with contaminated calibration blanks degrade precision and accuracy of the analyses performed by creating biases in target analyte calibration.

If the initial calibration blank is grossly negative for a target analyte, then the quantitation of that target analyte in the calibration standards will be biased high due to over compensation by the instrument. This will lead to low recovery issues with the CCV, ICV, field samples, and batch QC samples. If target analytes are present in the CALBLK leading the instrument to make an over correction of the baseline for these targets, then the calibration curve will be biased high yielding a low bias for those target analytes in the CCV, ICV, field samples, and batch QC.

10.8.2.2 Continuing calibration blank (ICB/CCB) is also analyzed following each initial and continuing calibration verification standard within an instrument sequence to verify instrument stability and system cleanliness. This does not baseline correct the instrument for possible contaminants in the background and must be evaluated to ensure that background corrections are appropriate and consistently applied throughout the sequence.

The ICB must not contain analytes > ½ RL, for CCB must not contain analytes >RL for all 6010 methods or, in the case of common laboratory contaminants, the concentrations should not exceed the RL. Common laboratory contaminants include: Calcium, Potassium, Magnesium, Zinc, Iron and Sodium.

NOTE: West Virginia requires all blank results to be less than the MDL. If the concentration of the target analyte is above the MDL, then corrective action and reanalysis is required, or the data must be qualified as including a potential high bias.

10.8.3 Rinse Blank

10.8.3.1 A rinse blank is utilized by the ICP to cleanse the system following the intake of each digestate analyzed. No data is obtained during this rinse and no applicable controls are required for this type of blank. This is merely cleansing the lines throughout the analytical system.

10.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - A matrix/matrix spike duplicate must be prepared for each matrix for each batch of 10 samples for method 200.7 or 20 samples for method 6010B/C/D, where sufficient sample volume was submitted by the client. Matrix spike and matrix spike duplicate are prepared from a sample aliquot spiked with the known concentration of analytes.

The matrix spike recoveries must meet the criteria in the table below unless the analyte concentration in the sample is at least four (4) times greater than the spike concentration.

Method	Acceptance Criteria	
	Water	Soil
6010B, C, and D	75 – 125%	75 – 125%
200.7	70-130%	NA

Assess that the matrix spike duplicate precision (%RPD) results meet project-established goals acceptance criteria. If no project goals are specified, then results for the RPD must be less than 20%.

10.10 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) - An LCS/LCSD pair must be digested and analyzed with each batch of 20 samples.

10.10.1 **For SW-846 Method 6010B, 6010C, and 6010D** – The LCS recovery must be 80-120%. The RPD must be less than 20%. When using a certified solid reference material for paint chip analysis, the manufacturer's established limits are used for control limits

10.10.2 **For EPA Method 200.7** – The LCS recovery must be 85-115%. The RPD must be less than 20%.

10.11 Interference Check Standards (ICSA/ICSAB/ICSA2/LA/CE) – The ICSA2, LA and CE must be analyzed at the beginning of each analytical run. The ICSA and ICSAB must be analyzed at the beginning and ending of each sequence or twice within each eight (8) hour shift, whichever is more frequent. The recovery of the ICSA and ICSAB elements must be

80-120%. If the results are unsatisfactory, see section 11.10 for further guidance. Do not proceed until this criterion is met.

NOTE: The unspiked elements in the ICSA, ICSA2, LA, and CE are not evaluated by the data capture software. The instrument analyst evaluates whether the unspiked elements are ±LLOQ for the ICSA, ICSA2, LA, and CE.

10.11.1 For Method 6010D two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup, and periodically (at least once every six (6) months) thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that and interference corrections applied are still valid.

10.11.1.1 Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 20mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 10mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 100mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 10mg/L.

The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be ±LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements.

The single element SIC checks are performed when the instrument is setup and periodically (at least once every six months) thereafter.

10.11.1.2 Mixed element interference check - The mixed element SIC solution (see section 7.10.3.2) is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than ±LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions (see Section 7.10.3.1). These may be present up to the concentration documented plus the LLOQ.

10.12 Serial Dilution (SD) - If the analyte concentration is sufficiently high (minimally 10X the IDL), an analysis of a 1:4 dilution must agree within 10% of the original determination. If not, see section 11.11 for further guidance.

For Method 6010D - If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within ±20% of the original determination.

10.13 Post digestion Spike (PS) - An analyte spike added to a portion of a prepared sample, or its dilution and must be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL. If the spike is not recovered within the specified limits, see section 11.11 for further guidance.

For Methods 6010C and 6010D – The Post Digestion Spike must recover within ±20% of the known value.

10.14 Internal Standard (ISTD) – Verify the internal standard responses. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample must fall within 60-140% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, see sections 9.15 & 11.9 for further guidance.

10.15 Sample Dilution - If a sample analyte concentration is quantitated within 90% or greater of the upper limit of the analyte Linear Dynamic Range (LDR), the sample must be diluted with acidified reagent water and re-analyzed.

10.16 Lower Limit of Quantitation (LLOQ) – When analyzing samples according to Method 6010D, the LLOQ is initially verified by the analysis of at least seven (7) replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

10.16.1 Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed, if needed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments should be noted when data is flagged.

11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038*, Data Review*.

11.3 Initial Calibration – After analyzing the calibration standards, the curve is reviewed to ensure the acceptance criteria described in section 10.5 are met. If analytes do not meet this requirement, corrective action must be taken. Corrective actions may include re-calibrating the instrument, replacing the tubing on the peristaltic pump, examining blanks and standards for degradation/contamination, or performing instrument maintenance. If the internal standard responses in the calibration standards do not meet the criteria in section 10.14, re-calibrate the instrument.

11.4 Initial Calibration Verification (ICV) – If the criteria described in section 10.6 are not met for a target analyte, re-analyze the ICV/ICVLL. If this fails a second time, corrective action must be taken. Re-calibrate and re-analyze the ICV/ICVLL using the same standard. If acceptance criteria are still not met, re-check standard curve and ICV/ICVLL preparation and/or perform routine instrument maintenance. If still not acceptable, refer to manufacturer's instruction or call service representative.

11.5 Continuing Calibration Verification (CCV) – The continuing calibration verification standard must agree with the criteria in section 10.7 or the CCV must be re-analyzed. If the recovery fails a second time, corrective action must be taken. The corrective action may require re-calibrate the instrument and re-analyze the last 10 samples, using the same CCV standard. If acceptance criteria are still not met, re-check the standard curve and CCV preparation and/or perform instrument maintenance. If the CCV still does not pass, refer to the manufacturer's instruction or call a service representative.

11.6 Blanks – Evaluate the blanks. The analyst must confirm that both the method blanks and the continuing calibration blanks were analyzed at the required frequency. Other items to check are as follows:

11.6.1 The instrument blank or continuing calibration blank (ICB/CCB) must meet the criteria in section 10.8. Corrective actions for method blank contamination include re-prepping the entire batch of samples, or if the site-specific requirements can be met, an elevated detection limit may be used, or the sample data qualified with a “B” qualifier and footnoted.

State Note: For West Virginia samples analyzed by Method 6010D, if the method blank fails to meet the necessary acceptance criteria, it should be re-analyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed, along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification
For West Virginia samples analyzed by Method 200.7, when blank values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of

contamination has been corrected and acceptable LRB values have been obtained.

NOTE: Method 6010D - If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification.

11.7 Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) – Assess that LCS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.10, rinse the instrument and re-analyze. If the LCS/LCSD fails for a second time, re-prepared all samples prepared in conjunction with the failing LCS. The affected samples must be re-digested and re-analyzed along with a new LCS. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client.

11.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) – Assess that MS pairs were prepared at the required frequency. If all target analyte recoveries are not within the criteria described in section 10.9, review the post digestion spike results for similar failures. Review the data to see if similar results are also present in the LCS/LCSD. If the LCS/LCSD are acceptable, the MS/MSD failures can be attributed to matrix interferences and the data can be qualified with a J4 and reported. If similar results are seen in the LCS, then re-prepared all samples prepared in conjunction with the failing MS/MSD. In this case, the affected samples must be re-digested and re-analyzed along with a new MS/MSD pair. If there is insufficient volume submitted to re-prepare the field samples, notify the project manager to contact the client for further instruction. Reporting with a qualifier may be performed if acceptable to the client. If insufficient field sample remains for re-analysis, report results with a J3 and an L3.

NOTE: If the sample concentration for an analyte is greater than four times (4x) the spike concentration, a “V” qualifier is used. The “V” qualifier indicates that the high concentration of analyte in the sample interfered with the ability to make an accurate spike recovery determination.

11.9 ISTD – The intensity of the Yttrium internal standard response in each sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in each sample must meet the criteria in section 10.14. If the %RI of the response in the sample falls outside of these limits, the laboratory must immediately re-analyze the calibration blank and monitor the internal standard intensities. If the %RI for that calibration blank is within the limits, the laboratory must re-analyze the original sample at a two-fold dilution due to a possible interference from the matrix on the ISTD. If the %RI for the re-analyzed calibration blank is outside the limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, the new calibration verified, and the samples reanalyzed.

11.10 Interference Check Standards (ICSA/ICSAB/ICSA2/LA/CE) - Evaluate the ICSA, ICSAB, ICSA2, LA, and CE. The analyst must verify that the ICS checks have been analyzed at the required frequency. If the criteria in section 10.11 are not met, check the background correction protocols currently in place for appropriateness. If these are the initial ICS checks run after daily calibration, re-analyze the CALBLANK and re-calibrate the instrument. If the ICSA and/or ICSAB did not agree at the end of an 8-hour shift, re-analyze the ICSA and ICSAB. If failure persists, perform instrument maintenance as needed, recalibrate and re-analyze any samples in the previous run that may have been affected.

11.11 Serial Dilution/Post-digestion Spike – The analyst must verify that the SD and PS have been analyzed at the required frequency. If either of these tests fails to meet the required criteria in sections 10.12 & 10.13, the possibility of a matrix interferent should be suspected. An O1 qualifier is used when either sample type fails due to matrix interferences.

11.11.1 Serial Dilution - An analysis of a 1:4 dilution must agree within 10% of the original determination. If not, a chemical or physical interference effect is suspected.

11.11.2 Post-digestion Spike - The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect is suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

11.12 Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.

11.12.1 If a method blank contains an amount of target analyte, but all samples are non-detected, the data may be reported with a “B3” flag. If a method blank contains an amount of target analyte, but the samples contain analyte at a level that is 10 times the level present in the method blanks, the data may be reported with a “B” flag.

11.12.2 If the MS/MSD fails (recovery less than 30% or greater than 150% and/or RPD greater than 30%) in an initial analysis and again upon re-analysis, the data is released with an appropriate qualifier as the failure is accepted as matrix related.

11.12.3 If a calibration verification standard is above the acceptable QC criteria and all samples being bracketed are below the reporting limit, the data is acceptable based on a high calibration bias with undetectable levels in the field samples. Any positive samples require re-analysis.

11.12.4 If the target analyte spiked in the quality control samples (LCS, LCSD, MS, MSD) exhibits high recovery and the target analytes in the field samples are below the reporting limit, the data may be released with a J+ qualifier indicating the high bias with no impact on the field sample analysis due to the bias present.

11.12.5 If the target analyte spiked into the QC pair (LCS/LCSD, MS/MSD) exhibit acceptable recoveries, but high calculated RPD values for precision, and the target analytes in the field sample are flagged with a J3 for the precision beyond acceptable quality control limits.

11.12.6 Sample results can be qualified and possible bias is narrated per the ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

11.12.7 Samples with multiple elements above the LDR must be diluted to verify interferences are not present

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that a laboratory waste management practice be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See ENV-SOP-MTJL-0051*, Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention*.

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

13.2 Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

13.3 Superscripts are provided where necessary to indicate the reference in Section 14.0 where the requirement/information can be found. Subscripts noted identify the most frequent/restrictive cases, but requirements may also be included at different frequencies/conditions in other references noted in section 14.0.

13.4 In the May 2012 Methods Update Rule, the EPA revised the previous interpretation of EPA 200.7 to include the use of axial torch orientation in the published method. Either axial or radial orientation is acceptable.

14.0 REFERENCES

14.1 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010B, Revision 2, December 1996.

14.2 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, EPA Method 200.7, Revision 4.4, May 1994.

14.3 *Identification of Test Procedures*, 40 CFR §136.3.

14.4 *Inorganic Chemical Sampling and Analytical Requirements*, 40 CFR §141.23.

14.5 *Inductively Coupled Plasma-Atomic Emission Spectrometry*, SW-846 Method 6010C, Revision 3, February, 2007.

14.6 *Hardness by Calculation*, Standard Methods 2340B, 20th Edition.

14.7 *Hardness by Calculation*, Standard Methods 2340B, 2011.

14.8 *Hardness by Calculation*, Standard Methods 2340B, 1997.

14.9 *Inorganic Analytes*, SW-846 Chapter 3, Revision 4, February, 2007.

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Attachment I: Revision History

Current Version (Pace National):

Date	Description of Revisions
8/13/2020	Technical and quality review and update. Revised header. Revised sections 2.7, 7.5, 7.11, 8.1.7, 10.3.2, 10.11, 11.8 and 11.10. Added sections 2.7.3, 2.7.4, 2.7.5, 7.11.3, 7.11.4, 7.11.5 and 7.12 and renumbered as necessary. Revised all subsections of 3.0.

Superseded Versions (ESC Lab Sciences SOP#340386):

Version	Date	Description of Revisions
0	5/1/95	Origination
1	7/25/95	
2	3/11/97	
3	8/18/99	
4	2/11/00	
5	8/21/00	
6	3/28/01	
7	12/14/01	
8	4/11/03	
9	1/26/04	
10	8/2/04	
11	10/15/05	Corrected CCV criteria for EPA 200.7
12	10/29/08	Technical and Quality Review and update. Corrected acceptance criteria in Section 10.6. Updated format and re-organized sections 8.0, 10.0 and 11.0 based on new format.
13	1/23/09	Technical and Quality Review and update.
14	2/2/09	Technical and Quality Review and update. Clarification of holding times, Inclusion of cross-references. Inclusion of section 13.1 and section 7.1.
15	4/15/11	Technical and Quality Review and update. Added state notes where applicable; Added Tables 1.2b & 1.2c; Revised Table 1.2a and Sections 1.1, 1.3, 1.6, 1.11, 2.18, 2.22, 5.6, 7.1, 7.6.2, 7.9, 8.1.3, 8.1.5, through 8.1.10, 9.1, 9.7 through 9.12, 10.3, 10.0 & 11.0, 12.1; Added Sections 2.13.1, 2.14.1, 2.31 through 2.35, 3.1.1, 4.1, 4.7, 7.17, 13.2, 13.3, 14.5 through 14.10.
16	6/27/14	Complete Rewrite and update.
17	12/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.13.1, 2.13.1, 2.23, 5.1.3, 6.1.1, 6.1.2, 7.1, 7.6, 7.7, 7.8, 7.9, 7.10.1, 7.10.2, 7.11, 7.12, 7.13, 7.14, 7.16, 8.1.3, 8.1.5, 8.1.11, 9.4, 10.2, 10.3.1, 10.3.2, 10.4.2, 10.4.4, 10.9, 10.10, 10.14, 11.12.6, and 12.2. Revised Tables 1.2a, and 1.10, Deleted Sections 2.22 and 7.12. Added Attachment III.

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ISSUER: Pace National – Mt. Juliet, Tennessee

Version	Date	Description of Revisions
18	10/28/2016	Technical and quality review and update. Update per South Carolina DHEC correspondence of 6/24/16. Header and signature block re-formatting. Revised SOP title. Revised Sections 1.1, Table 1.2a, 1.3, 1.6, 1.10, 2.12, 2.15, 5.6, 7.5.2, 7.10.1, 9.1, 10.2, 10.3.1, 10.4.2, 10.4.3, 10.4.5, 10.5, 10.6.1, 10.6.2, 10.7.1, 10.8.1.2, 10.8.2.2, 10.10.1, 10.16, and Attachment III Table 2. Deleted Table 1.2b. Deleted Sections 2.2, 2.12, 2.13, 2.14, 2.15, 2.17, 2.20, through 2.33, 3.5, 4.8, 7.14, 7.16, 8.1.3, 9.7 through 9.10, 9.13, 10.4.5.1, 10.4.5.2, 10.4.5.3, 10.10.3, 13.5, 14.7, 14.8, 14.9, and 14.10. Added Sections 2.14, 7.10.3 and all subsections, 9.11, 9.12, 10.1.1, 10.6.4, 10.6.5, 10.8.1.2, 10.9.1 and all subsections, 10.11.1 and all subsections, and 11.6.1.
19	11/30/2017	Update in response to A2LA audit finding CAR2872. Changed ESC logo. Updated Sections 1.5, 3.1, 7.9, 10.11, 14.1, 14.2, 14.3, 14.4, 14.5, 14.6, 14.7, 14.8, 14.9, and Attachment III Table 5.
20	7/27/18	Update in response to WI and AZ audit findings. Changed logo and references to "ESC" to "Pace National". Revised Section 7.14, 10.4.4, and 10.7.3. Added State Note to Sections 10.8.1.2 and 10.11. Also added 6010C note to Section 10.13
21	9/10/18	Update in response AZ audit finding CAR3296. Revised Post-Spike acceptance criteria to ±20% in Section 10.9.1.2

Superseded Versions (Pace National):

Date	Description of Revisions
1/27/2019	Technical and quality review and update. Deleted header, footer and signature. Revised sections 1.0, 1.3, 1.5, 1.6, 1.7, 1.13, 1.13.1, 2.8.1(2.7.1), 2.8.2(2.7.1), 4.2, 4.4, 4.7, 6.2, 6.3, 6.4, 6.5, 6.6, 7.1, 7.2, 7.3, 7.4, 7.5.1, 7.5.3, 7.6, 7.7, 7.8, 7.9, 7.10, 7.10.1, 7.10.2, 7.13, 9.5, 9.6, 9.9, 10.1, 10.2, 10.3, 10.4, 10.4.3, 10.4.5, 10.5, 10.6.3, 10.6.4, 10.6.5, 10.7.3, 10.8, 10.8.1, 10.8.1.2, 10.8.2, 10.8.3, 10.10.1, 10.11, 10.11.1.2, 10.12, 10.13, 10.16, 11.1, 11.2, 11.12.6, 12.1, 12.2, 14.2, 14.6, 14.7 and 14.8. Revised Table 1.10. Added section 7.5 and renumbered as necessary. Deleted sections 7.10.3, 7.10.3.1, 7.10.3.2, 7.11, 7.12, 7.14, 10.9.1, 10.9.1.1 and 10.9.1.2. Revised Attachment I. Revised Attachment II sections 4.10, 4.17 and 4.18.
5/7/2019	Technical and quality review and update. Revised sections 1.1, 1.2, 2.14, 8.1.1.2, 10.3.2, 10.9, 10.11, 10.11.1, 10.11.1.1, 10.11.1.2, 11.8, 11.12.6 and 12.1. Correctly numbered section 7.6.1.1.
6/19/2019	Added corporate header and footer. Revised based on LA DW Auditor's comments. Revised Sections 8.1.6, 10.3.2, and Attachment I.
10/16/2019	Revised section 1.2 and 10.7.1. Added section 11.12.7.
2/4/2020	West Virginia audit response. Revised Section 10.8.2.2.
4/8/2020	Revision in response to West Virginia audit deficiency. Revised Sections 10.8.1.2 and 11.6.1.

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Attachment II: Potential ICP interferences arising from analytes present in field samples at concentrations of 100mg/L

Analyte	Wavelength (nm)	Interferant[a,b] Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 mg/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.
[c] Interferences will be affected by background choice and other interferences may be present.

NOTE: Using the above table, if analyzing for Lead in a sample containing 1000mg/L Aluminum, the lead results could demonstrate a high bias of 0.17mg/L. (If the sample contained 10000mg/L of Al, the bias in lead could be 1.7mg/L), if background corrections are not accurately applied by the instrument.

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Attachment III: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing pump tubing, replacing the torch, cleaning the nebulizer, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

QTegra, Version 2.4

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
Poor Precision	Nebulizer Pressure	Pressure should be about 0.15 mPa for aqueous solutions. If pressure is substantially higher, clean the nebulizer orifice or replace it entirely.
	Pooling in Spray Chamber	Usually caused by an oily film in the spray chamber. Aspirate 0.1% HF solution for about 20 seconds or 0.01% Triton X-100 solution.
	Center Tube	Replace the tube.
	Capillary Tubing	Air bubble migration through tubing should be smooth and consistent. Replace kinked/ pinched tubing.
	Peristaltic Pump	Adjust platen pressure. Check for leaks. Replace damaged pump.
Poor Accuracy	Pump Rate	Ensure the flush pump rate is the same as the analysis pump rate.
	Flush Time	Ensure proper time set for adequate rinse (typically 30 seconds).
Poor Detection Limits	Dirty Window or Mirror	Clean or replace dirty components.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction

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factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is th volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature

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Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.6 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a

measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.

+ If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.

+ The laboratory shall maintain documentation for all MDL determinations and LOD verifications.

+ The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.12 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

+ If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

+ Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.

+ If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.

+ Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.

+ Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.18 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.19 The method blank shall be considered to be contaminated if:

+ The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;

+ The concentration of any common laboratory contaminant in the blank exceeds the LOQ;

+ If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

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Table 3. LCS Control Limits – Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

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Table 4. LCS Control Limits – Method 6010 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since the last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLCCV). Low-level calibration check standard should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 and 4 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.

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Table 5. Quality Control Requirements – Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

Appendix F: Quality Control Forms

320-1 SITE CONTROL LOG (SCL)

Contract Number:		**Task Order No:**	
CAPE Project Number:		**Date:**	
Project Location:			
Project Description:			

TIME		NAME	ORGANIZATION
IN	OUT		

320-2 DAILY QUALITY CONTROL REPORT

Daily Report Number:			Day:		Date:	

Project Title:					Contract No.:	
					Task Order No.:	
Weather:	☐ Clear	☐ Partly Cloudy	☐ Cloudy	Temperature:	Min.	Max.
Wind:	☐ Calm	☐ Breeze	☐ Windy	Precipitation:	Rain	Snow
				Weather Information Source:	www.accuweather.com	

1. Labor Summary - Contractor & Subcontractor Supervision and Craft Personnel onsite and Area of Responsibility:

Number	Labor Category	Hours	Cumulative Hours	Employer	Area of Responsibility
CAPE Supervision					
1				CAPE	
2				CAPE	
3				CAPE	
CAPE Craft					
4				CAPE	
5				CAPE	
6				CAPE	
7				CAPE	
Subcontractor(s) Supervision					
8					
9					
10					
11					
Subcontractor(s) Craft					
12					
13					
14					
15					
	Total Hours:	0	0		

Comments (List any Visitors to Project and purpose of Visit):

2. Equipment (Not Hand Tools): shaded items indicate equipment that has been taken off rent and/or offsite

Description (Make and Model Number)	Arrival Date	Departure Date	Date of Last Safety Check	Days on Rent	Hours Used	Hours Idle	Hours Repair

Comments:

3. Work Performed Today: (Indicate location and description of work performed by CAPE and/or Subcontractors. When network analysis is used, identify work by activity number)

a	
b	
c	
d	

320-2 DAILY QUALITY CONTROL REPORT

Daily Report Number:			Day:		Date:	

e	
f	

4. Three Phase Control Activities Performed:

Definable Features of Work (DFW)		Meetings / Inspections Completed		
		Preparatory	Initial	Follow-up
a				
b				
c				
d				
e				
f				

5. Submittals Reviewed:

Submittal Number	Specification / Plan Reference	Reviewed By:	Action by Government (FIO or GA)	Approval Received: (Date)

6. Tests Performed and Test Results:

Laboratory Analytical Testing:

Type of Sample	Sample Date	Matrix	Sample ID No.	Analyses Requested	Comments

Geotechnical and Material Testing:

Type of Testing Performed	Test Date	Results of Testing	Comments

Field Screening:

Type of Testing Performed	Test Date	Results of Testing	Comments

Comments:

7. Inspections Performed and Inspection Results:

Area / Work Element Inspected	Location of Inspection on Project Site	Inspection Results (Accept / Reject)

8. Material Received: (Note Inspection results and storage provided)

Item	Description	Unit of Measure	Daily Quantity	Cumulative Quantity	Storage Provided	Inspection Results (Accept or Reject)	Complies with Buy American Act
a							☐ Yes ☐ No
b							☐ Yes ☐ No
							☐ Yes ☐ No

320-2 DAILY QUALITY CONTROL REPORT

Daily Report Number:			Day:		Date:	

c							☐ Yes ☐ No
d							☐ Yes ☐ No

9. Offsite Surveillance Activities (visits to suppliers, fabricators, quarries, machining facilities, etc):

Supplier or Facility Visited	Supplier Representative Name	Product Supplied	Results of Visit

10. Transportation and Disposal of Liquids, Solids, Recyclable Steel, and Government Property:

Non-Hazardous Transportation and Disposal

Waste Type	Daily Volume	Cumulative Total	Transporter	Disposal Facility

Recyclable Material Transportation and Management

Material Type	Daily Volume	Cumulative Total	Transporter	Receiving Facility

Government Property Management

Description	Date of Disposition	Receiving Agency / Facility

11. Job Safety: (List items checked, results, instructions, and corrective actions taken)

Inspections Conducted:

Heavy Equipment		Vehicles		Electrical Chords		First Aid Kit	
Lifting Straps / Cables		Fire Extinguishers		Flammables		Power Tools	
Personnel PPE		Work Zone Barriers		Overhead Lines			

Comments: include violations, corrective measures, damaged or compromised equipment, etc.

a	
b	
c	
d	

Daily Tailgate Safety Meeting: (summarize topics discussed)

Were all activities conducted in accordance with EM 385-1-1?		Yes	

12. Remarks: (Instructions received or given. Conflicts in Plans and/or Specifications. Delays encountered)

13. Planned Activities: (List anticipated field activities for next day of work)

14. Safety Hours:

Daily safety hours including CAPE and Subcontractors:			Number of On-site Workdays:	
Cumulative safety hours to date:			Calendar Days since Start of Work:	

Contractor's Verification: On behalf of the Contractor, I certify that this report is complete and correct, and all materials and equipment used and work performed during this reporting period are in compliance with the contract plans and specifications, to the best of my knowledge, except as may be noted above.

320-2 DAILY QUALITY CONTROL REPORT

Daily Report Number:			Day:		Date:	

CQC System Manager / Initials

Date

Site Supervisor / Initials

Date

320-3 FIELD CHANGE REQUEST (FCR)

Contract Number:		**Task Order No:**	
CAPE Project Number:		**Date:**	
Project Location:		**FCR No.:**	
Project Description:			

CHANGE IN: (Check One)	**IMPACT:** (Check all that apply)	**ACTION REQUIRED** (Check all that apply)
☐ A Field Condition ☐ B Construction / Production ☐ C Other, Explain	☐ Scope of Work (SOW) ☐ Schedule ☐ Budget ☐ Approved Plans and Documents ☐ Other, Explain	☐ Review of Cost Impact ☐ Update Schedule to Reflect Change ☐ Review/Approval by Project Manager ☐ Review/Approval by Client Representative ☐ Submit Request for Modification ☐ Other, Explain
Explanation:	Explanation:	Explanation:

1. CHANGE REQUEST INFORMATION:

1. Description of Needed Change: **Sketch Attached:** ☐ Yes ☐ No

2. Reason for Needed Change:

3. Consequences of Failure to Implement Change:

4. Other work that will be affected by the Change:

2. SIGNATURES AND DATE:

CAPE QC OFFICER		**DATE**	
CAPE PROJECT MANAGER		**DATE**	
CAPE SITE SUPERINTENDENT		**DATE**	
CAPE PROJECT ENGINEER		**DATE**	
CLIENT REPRESENTATIVE		**DATE**	

320-4 NONCONFORMANCE REPORT (NCR)

Contract Number:		Task Order No:	
CAPE Project Number:		Date:	
Project Location:		NCR No.	
Project Description:			

1. NONCONFORMANCE DESCRIPTION:			
IDENTIFIED BY:		DATE	

2. CORRECTIVE ACTION REQUIRED TO RECTIFY AND PREVENT RECURRENCE:			
PREPARED BY:		DATE	
TO BE PERFORMED BY:		DATE	
TO BE VERIFIED BY:		DATE	

3. CORRECTIVE ACTION TAKEN / COMPLETED:			
PERFORMED BY:		DATE	
VERIFIED BY:		DATE	

4. REMARKS:

320-5 PREPARATORY PHASE INSPECTION / MEETING

Contract Number:		Task Order No:			
CAPE Project No.:		Date:			
Project Location:					
DFW:		Spec Sect.		Date	

1. PREPARATORY PHASE INSPECTION / MEETING ATTENDEES:

	NAME	POSITION	COMPANY / CLIENT
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

2. PREPARATORY PHASE INSPECTION CHECKLIST:

A. DOCUMENT REVIEW:

Done	N/A	Description	Results	Action Items
☐	☐	Review each paragraph of applicable specifications and all related drawings.		
☐	☐	Review standards and procedures referenced on drawings and in specifications.		
☐	☐	Review of the Project Shop Drawings.		

B. SUBMITTAL STATUS REVIEW:

Review of all Submittal requirements to ensure that all materials and/or equipment have been tested, submitted, and approved.

☐	☐	Have all materials been submitted, tested, and approved?	☐ Yes ☐ No	
☐	☐	Has all equipment been submitted, tested, and approved?	☐ Yes ☐ No	

C. OFFSITE DISPOSAL OF MATERIALS:

☐	☐	Have all materials for disposal offsite been sampled and properly characterized for disposal?	☐ Yes ☐ No ☐ N/A	
☐	☐	Have Landfills been contacted and received copy of waste characterization results?	☐ Yes ☐ No ☐ N/A	

320-5 PREPARATORY PHASE INSPECTION / MEETING

Contract Number:		**Task Order No:**			
CAPE Project No.:		**Date:**			
Project Location:					
DFW:		**Spec Sect.**		**Date**	

☐	☐	Have all Parties related to the approval process for hauling offsite been contacted and have they approved disposal methods?	☐ Yes ☐ No ☐ N/A	
☐	☐	Has it been verified that the Transporter of the material is properly licensed for hauling of this material?	☐ Yes ☐ No ☐ N/A	
☐	☐	Has the process/procedure for signing Waste Manifests been clearly established?	☐ Yes ☐ No ☐ N/A	
D. WORK AREA INSPECTION:				
☐	☐	Has all required preliminary work been completed and accepted to allow this DFW to start?	☐ Yes ☐ No ☐ N/A	
☐	☐	Are all required Permits received and on file on the jobsite and/or properly posted?	☐ Yes ☐ No ☐ N/A	
E. MATERIAL AND EQUIPMENT INSPECTION:				
☐	☐	Are all required materials on hand (or scheduled for delivery to avoid schedule delays)?	☐ Yes ☐ No ☐ N/A	
☐	☐	Is all material properly stored and protected, as applicable?	☐ Yes ☐ No ☐ N/A	
☐	☐	Are all pieces of equipment or modules on hand (or scheduled for delivery to avoid schedule delays)?	☐ Yes ☐ No ☐ N/A	
☐	☐	Is all equipment or modules properly stored and protected, as applicable?	☐ Yes ☐ No ☐ N/A	
F. REVIEW OF SAFETY REQUIREMENTS:				
☐	☐	*Review approriate AHAs to ensure safety requirements are met.* Comments:		

320-5 PREPARATORY PHASE INSPECTION / MEETING

Contract Number:		**Task Order No:**			
CAPE Project No.:		**Date:**			
Project Location:					
DFW:		**Spec Sect.**		**Date**	

G. REVIEW OF WORK PERFORMANCE / TESTING / INSPECTION REQUIREMENTS:				
☐	☐	Discuss procedures to accomplish the work, including points of control.		
☐	☐	Establish construction tolerances and workmanship standards for this DFW.		
☐	☐	Review provisions that have been made to provide required quality control (check applicable one)		
		☐ Subcontractor / Consultant		
		☐ QC Officer or another member of QC Team		
		☐ 3rd Party Inspection		
☐	☐	Quality Control Testing:		
		Tests to be Performed:		
		Frequency of Tests:		
		Testing by Whom:		
		When:		
		Where:		
		Has Testing Facility Been Approved?	☐ Yes ☐ No	
		For Testing performed on site, has testing equipment and test methods been submitted?	☐ Yes ☐ No	
		Has Testing Equipment been calibrated before use or calibration certificate been provided before use?	☐ Yes ☐ No	
☐	☐	Verify that portion of Work Plan for work to be performed has been accepted by the government.		
☐	☐	Discuss the Initial Control Phase.		

I hereby declare that: The above required materials delivered to the job site and methods and procedures are certified to fully comply with the project requirements.

Quality Control Representative: _____

320-6 INITIAL PHASE INSPECTION / MEETING

Contract Number:		**Task Order No:**			
CAPE Project No.:		**Date:**			
Project Location:					
DFW:		**Spec Sect.**		**Date**	

1. INITIAL PHASE INSPECTION / MEETING ATTENDEES:

	NAME	POSITION	COMPANY / CLIENT
1			
2			
3			
4			
5			
Was Client Representative notified?			☐ Yes ☐ No

2. INITIAL PHASE INSPECTION CHECKLIST:

A. GENERAL ITEMS:

Done	N/A	Description	Results	Action Items
☐	☐	Check preliminary work and review minutes of the Preparatory Inspection / Meeting.		
☐	☐	Check that materials and equipment being used comply with project requirements.		
☐	☐	Check the work to ensure it is full compliance with the project requirements.		

B. CONTROLS TO ASSURE FULL COMPLIANCE:

		Controls	Testing
☐	☐	☐ QC Officer Observations ☐ Qualified Inspector ☐ 3rd Party Inspection & Testing ☐ Other, _____	☐ Checked Testing procedure ☐ Checked Instrumentation Calibration ☐ Checked recording Forms & Tracking ID No. ☐ None

C. ESTABLISH LEVEL OF WORKMANSHIP:

☐	☐	Work Location:		
		Is a sample panel required?	☐ Yes	☐ No
		Is initial work considered as a sample?	☐ Yes	☐ No
☐	☐	Check for omissions and resolve any differences or interpretations with the government/client representative.		
☐	☐	Check safety to include compliance with Safety Plan and Activity Hazard Analyses. Review the Activity Hazard Analyses.		
☐	☐	Were procedures and work methods witnessed in strict compliance with project requirements?	☐ Yes ☐ No	
☐	☐	Is a re-inspection required?	☐ Yes ☐ No	

D. BRIEF SUMMARY OF INITIAL INSPECTION PROCEDURE AND RESULT, POINTS OF CONCERN, ETC.:

Quality Control Representative: _____

320-7 PRE-FINAL INSPECTION / PUNCHLIST

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			
Specific Area of Inspection:			

1. PRE-FINAL INSPECTION PARTICIPANTS:

	NAME	POSITION	COMPANY / CLIENT
1			
2			
3			
4			
5			
Was Client Representative notified?			☐ Yes ☐ No

2. BRIEF DESCRIPTION OF WORK PERFORMED AS DESCRIBED IN TASK ORDER SOW:

3. TYPE OF INSPECTION:

☐ Visual Inspection of the Work
☐ Documentation Review

Note: As part of the Pre-Final Inspection, status of all requirements of Task Order must be reviewed - as-built drwgs, O&M Manuals, Warranties, etc.

4. REFERENCE DOCUMENTS (CHECK ALL THAT APPLY):

☐ Drawings ☐ Work Plans
☐ Specifications ☐ Other _____

5. FINDINGS (LIST ALL ITEMS NOT CONFORMING TO REQUIREMENTS OF REFERENCE DOCUMENTS):

Item	Description of Deficiency	Reference Document	Estimated Date of Completion
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			

Quality Control Representative: _____

320-8 FINAL ACCEPTANCE INSPECTION

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			
Specific Area of Inspection:			

1. FINAL INSPECTION PARTICIPANTS:

	NAME	POSITION	COMPANY / CLIENT
1			
2			
3			
4			
5			
6			

2. BRIEF DESCRIPTION OF WORK PERFORMED AS DESCRIBED IN TASK ORDER SOW:

3. TYPE OF INSPECTION:

- ☐ Visual Inspection of the Work
- ☐ Documentation Review

4. REFERENCE DOCUMENTS (CHECK ALL THAT APPLY):

- ☐ Drawings
- ☐ Specifications
- ☐ Work Plans
- ☐ Other _____

5. FINDINGS:

6. REMARKS:

- ☐ Project meets SOW Requirements
- ☐ Project does not meet all SOW Requirements and a Re-Inspection must be scheduled.

Date Scheduled for Re-Inspection: _____

Additional Remarks:

Quality Control Representative: _____

320-10 FIELD INSPECTION REPORT

Contract Number:		**Task Order No:**			
CAPE Project No.:		**Date:**			
Project Location:					
DFW:		**Spec Sect.**		**Date**	
Inspection Performed by:					

1. DESCRIPTION OF WORK INSPECTED:

2. TYPE OF INSPECTION:

- ☐ Visual
- ☐ Dimensional
- ☐ Non-Destructive Examination (NDE)
- ☐ Other _____
- ☐ In Progress
- ☐ Final

3. REFERENCED DRAWINGS / STANDARDS:

4. FINDINGS:

5. SKETCH, IF APPLICABLE:

Inspector: _____

Quality Control Representative: _____

320-11 FIELD TESTING REPORT

Contract Number:		Task Order No:			
CAPE Project No.:		Date:			
Project Location:					
DFW:		Spec Sect.		Date	
Testing Performed by:					

1. DESCRIPTION OF WORK TESTED:			
Testing Equipment Used:		Latest Calibration Date:	

2. TYPE OF TESTING PERFORMED:

Test Number	Type of Test	Test Duration, if Applicable	Specification Requirement(s)	Test Results	Pass / Fail

☐ In Progress ☐ Final

3. REFERENCED DRAWINGS / STANDARDS:

4. COMMENTS:

5. SKETCH, IF APPLICABLE:

Testing Individual: _____

Quality Control Representative: _____

320-12 LIST OF OUTSTANDING DEFICIENCIES

Contract Number:		**Task Order No:**	
CAPE Project No.:		**CQC Report No.:**	
Project Location:			

Spec Section, Drwg No., or SOW Reference	Location on Project	Deficiency Description	Date Found	Date to be Corrected	Date Corrected	Remarks

Note: This form shall be used by the Contractor to track outstanding construction deficiencies.

Quality Control Representative: _____

320-13 RECORD OF PREPARATORY AND INITIAL INSPECTIONS

Contract Number:		Task Order No:	
CAPE Project No.:			
Project Location:			

Date of Inspection	Type of Inspection (Preparatory or Initial)	Description of Definable Feature of Work (DFW)	Reference Daily Quality Control Report (DQCR) Number	Remarks

320-14 - PHOTO LOG

Contract Number:		**Task Order No:**	
CAPE Project No.:			
Project Location:			

Photo Number	Date Taken	Description

320-14A - PHOTO RECORD

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			

PHOTO NUMBER:		**DATE TAKEN:**	
Description:			

PHOTO NUMBER:		**DATE TAKEN:**	
Description:			

320-15 WASTE MANIFEST SUMMARY

Contract Number		DO/TO No:		CAPE Project #			Project Location	

Destination of Waste	*Enter Landfill Name and Address*
Waste Generation Site	*Enter Site Name and Location from Which Material was Generated*
Process Generating Waste	*Enter the process generating the waste, ie Building Demolition Debris, Excavation of contaminated soils, etc.*

Load Number	Date	Time Out	Transporter	Manifest Number	Transporter Ticket Number	Subcontractor Ticket Number (if applicable)	Waste Type	Solids (Tons) or Liquids (gallons)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
			TOTAL DISPOSAL QUANTITY (tons or gallons)					0.00

320-16 WASTE INVENTORY SUMMARY

Contract Number		DO/TO No:		CAPE Project #			Project Location	
Process Generating Waste	*Enter the process generating the waste, ie Building Demolition Debris, Excavation of contaminated soils, etc.*							

Load Number	Date	Description	Quantity (TNS or GAL)	Waste Classificatiion	Receiving Disposal Facility	Method of Disposal	Date of Disposal or Destruction
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31							
32							
		TOTAL QUANTITY:	**0.00**				

320-17 PROJECT DOCUMENT CONTROL LOG

Contract Number:		**Task Order No:**	
CAPE Project No.:			
Project Location:			

Item No.	Document Identification (Drwg. No.; Spec. Section, etc.)	Original Revision Number	Document Title or Description	Current Revision Number if Revision Issued	Date of Latest Revision	List of Personnel, Subcontractors, or Vendors who have been issued this document	Revision Issued to all Holders	Old Versions Returned
1							☐	☐
2							☐	☐
3							☐	☐
4							☐	☐
5							☐	☐
6							☐	☐
7							☐	☐
8							☐	☐
9							☐	☐
10							☐	☐
11							☐	☐
12							☐	☐
13							☐	☐
14							☐	☐
15							☐	☐

320-17 PROJECT DOCUMENT CONTROL LOG

Contract Number:		**Task Order No:**	
CAPE Project No.:			
Project Location:			

Item No.	Document Identification (Drwg. No.; Spec. Section, etc.)	Original Revision Number	Document Title or Description	Current Revision Number if Revision Issued	Date of Latest Revision	List of Personnel, Subcontractors, or Vendors who have been issued this document	Revision Issued to all Holders	Old Versions Returned
16							☐	☐
17							☐	☐
18							☐	☐
19							☐	☐
20							☐	☐
21							☐	☐
22							☐	☐
23							☐	☐
24							☐	☐
25							☐	☐
26							☐	☐
27							☐	☐
28							☐	☐

Quality Control Representative: _____

329-1 REWORK DATA COLLECTION FORM

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			
Originator:		**Rework Tracking Number:**	

QUALITY INCIDENT DESCRIPTION:

ROOT CAUSE CLASSIFICATION:					
Category	**Contributing Cause**				**Direct Cause**
Design / Deliverable Preparation	1	01	☐	Poor document control	
		02	☐	Constructability Problems	
		03	☐	Errors and Omissions	
		04	☐	Inadequate or defective design	
		05	☐	Error in equipment or material selection	
Field Execution	2	01	☐	Work Organizatiion/Planning deficiency	
		02	☐	Unrealistic schedule	
		03	☐	Inadequate supervision	
		04	☐	Improper resource allocation	
		05	☐	Policy not adequately defined, disseminated, or enforced	
		06	☐	Other management problems	
Material and equipment	3	01	☐	Untimely deliveries	
		02	☐	Prefabrication, construction, or equipment not to Project requirements	
		03	☐	Non-compliance with Specifications	
		04	☐	Materials not in right place when needed	
		05	☐	Contamination	

329-1 REWORK DATA COLLECTION FORM

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			
Originator:		**Rework Tracking Number:**	

People	**4**	**01**	☐	Insufficient knowledge / skill levels	
		02	☐	Lack of training	
		03	☐	Lack of resources	
		04	☐	Lack of procedures / Inadequate procedures	
		05	☐	Other human error	
PJM - Leadership & Communications	**5**	**01**	☐	Inadequate supervision	
		02	☐	Lack of employee buy-in	
		03	☐	Lack of safety and QA/QC commitment	
		04	☐	Poor communication / unclear instructions to employees	
		05	☐	Inadequate job planning	
		06	☐	Lack of client management	

COLLECT PHYSICAL EVIDENCE / SUMMARY OF FACTS / PHOTOS, ETC.:	
A. Physical Evidence:	
1. Environmental Impact on Conditions	☐ NA
a) Weather:	
b) Work Conditions:	
2. Photos or Sketches: (*attach as applicablef)*	☐ NA
3. Other:	☐ NA
B. Witness Statements: Summary of Facts	
C. Document Review: Summary of Facts	

329-1 REWORK DATA COLLECTION FORM

Contract Number:		**Task Order No:**	
CAPE Project No.:		**Date:**	
Project Location:			
Originator:		**Rework Tracking Number:**	

ANALYSIS:

1. Was there a procedure violation? ☐ Yes ☐ No
   If yes, what was unclear or incorrect?
2. Is this a recurring incident? ☐ Yes ☐ No
3. Was construction Health & Safety Plan violated? ☐ Yes ☐ No
4. Was an Permit violated? ☐ Yes ☐ No
5. Other: _____

Prepared by: _____ **Date:** _____

QC Officer: _____ **Date:** _____

Project Manager: _____ **Date:** _____

COST CONTROL:

Attach copy of latest Project Status Report for costs associated with this Rework:

QUALITY COUNCIL:

Applicable Cause Code:	Contributing Cause

Root Cause

CORRECTIVE ACTION:

QC Manager: _____ **Date:** _____

General Manager: _____ **Date:** _____

372-1 Field Quality Control Performance Audit

Contract Number:		Task Order No:	
CAPE Project Number:		Date:	
Project Location:			
Project Description:			
Project Manager:		CQCSM:	
Site Superintendent:			

	Yes	No	N.A.	Date Corrected
SITE QUALITY CONTROL STAFF				
1. Is Documentation of Proper Quality Control Training on File?	☐	☐	☐	
2. Letter of Authority on Site for CQC System Manager (CQCSM)?	☐	☐	☐	
3. Are there additional QC Staff on the Project?	☐	☐	☐	
4. If No. 3 is "Yes", is Documentation of Training on File?	☐	☐	☐	
5. If No. 3 is "Yes", do Qualifications meet requirements of Specs?	☐	☐	☐	
QUALITY CONTROL FILES				
1. Are QC Files well organized for ease of finding QC documents?	☐	☐	☐	
2. Is there a copy of the Contract Documents on site?	☐	☐	☐	
PROJECT PLANS				
1. Are copies of all Project Plans available on Site?	☐	☐	☐	
2. Is documentation of any Plan Modifications on File?	☐	☐	☐	
3. Is documentation of review of Plans by Site Superintendent/Staff on File?	☐	☐	☐	
4. Is there documentation of review of Project QC Requirements with Site Superintendent and Site Staff?	☐	☐	☐	
PROJECT DESIGN				
1. Does the Project include a Design element for which CAPE is responsible?	☐	☐	☐	
2. If "Yes", is there a Design Quality Control Plan?	☐	☐	☐	
3. Is there a CAPE Design Quality Control Manager assigned?	☐	☐	☐	
SUBMITTALS				
1. Is there a Submittal Register being used on the project?	☐	☐	☐	
2. Are all Submittals approved prior to the start of any work relating to the material on the Submittal Register?	☐	☐	☐	
CONTROL: THREE-PHASE INSPECTION PROCESS				
1. Is Documentation of Three-Phase Inspections/Meetings on File? Preparatory Phase - documented preparatory phase inspection checklist including verification that all required permits, materials, notifications, etc. are in place prior to start of work for each DFW	☐	☐	☐	

372-1 Field Quality Control Performance Audit

Contract Number:		Task Order No:	
CAPE Project Number:		Date:	
Project Location:			
Project Description:			
Project Manager:		CQCSM:	
Site Superintendent:			

	Yes	No	N.A.	Date Corrected
Initial Phase - documented initial phase inspection checklist indicating that the procedures are actually being followed in the field for each DFW	☐	☐	☐	
Follow up Phase - Documented follow up inspection of work performed in the Daily Quality Control Report?	☐	☐	☐	
2. If field work is complete, is there documentation of the Pre-final Inspection?	☐	☐	☐	
Is there documentation of the Punch list inspection?	☐	☐	☐	
Is there documentation of the Final Acceptance inspection?	☐	☐	☐	
3. Are personnel doing Daily Inspections qualified and knowledgeable of the work that they are responsible for inspecting?	☐	☐	☐	
TESTING: QUALITY CONTROL PERFORMANCE				
1. Is there a Summary of all Testing Requirements for the Project available on site?	☐	☐	☐	
2. Is documentation of all Testing and Test Results on file?	☐	☐	☐	
3. Are all Testing Laboratories being used properly certified per client or CAPE requirements with documentation on file?	☐	☐	☐	
4. Is there documentation on file of all corrections to any Tests that did not pass initial testing?	☐	☐	☐	
DOCUMENTATION: QUALITY CONTROL REPORTS				
1. Are Daily Quality Control Reports (DQCR) up to date?	☐	☐	☐	
2. Have Daily Quality Control Reports been certified by the CQCSM?	☐	☐	☐	
3. Are Reports being completed on CAPE Standard Daily QC Report Form?	☐	☐	☐	
4. Are the outstanding deficiencies being documented and tracked to correction?	☐	☐	☐	
5. Does the DQCR document the daily safety meeting including the topic discussed today?	☐	☐	☐	

372-1 Field Quality Control Performance Audit

Contract Number:		Task Order No:	
CAPE Project Number:		Date:	
Project Location:			
Project Description:			
Project Manager:		CQCSM:	
Site Superintendent:			

	Yes	No	N.A.	Date Corrected
DOCUMENTATION: DRAWINGS AND SPECIFICATIONS				
1. Is there documentation as to the holders of all drawings and specifications related to the project, ie CAPE and any subcontractors?	☐	☐	☐	
2. Is there documentation that shows that all holders of drawings and specifications have been provided any/all revisions such that all personnel, including subs, are working with the most current versions of all documents?	☐	☐	☐	
3. Are As-Builts being maintained on the Site and are they up-to-date?	☐	☐	☐	

AUDIT PERFORMED BY: _____ DATE: _____

POSITION / TITLE: _____

CQC SUPERVISOR: _____ DATE: _____

PROJECT MANAGER: _____ DATE: _____

AUDIT APPROVAL: _____ DATE: _____

Quality Control Manager - R&ICON BU

AUDIT NOTES OR COMMENTS

380-1 ARCHIVING REGISTRATION FORM

BOX Number:			
Contract Number:		**Task Order No:**	
CAPE Project No.:			
Contents Description:			
Organization:		**Department:**	

File / Reference No.	Date	Document Description / File Title	Remarks

Originator	**Name**	
	Signature	
Dates	**Archiving**	
	Destruction	

MATERIAL APPROVAL SUBMITTAL

Form Approval
OMB No 9000-0062

TO ***(Contracting Officer)***	**FROM** ***(Contractor)*** CAPE Environmental Management Inc.	**DATE**
CONTRACT NUMBER	**TRANSMISSION NUMBER**	**SUBMITTAL** NEW / RESUBMITTAL
PREVIOUS SUBMISSION NUMBER	**PROJECT NUMBER**	

TO BE COMPLETED BY CONTRACTOR													**FOR GOVERNMENT USE ONLY**			
			NO. OF COPIES REQU RED													
LINE NUMBER	SPEC FICAT ION SECTION/ PARA NO /DRAWI NG NO.	DESCRIPTION OF MATERIAL	CERTIFICAT ION OF COMPLIAN CE	SHOP DRAWINGS	SAMPLES	COLOR SELECTION	MANUFACT URER'S RECOMME NDATIONS	MANUFACT URER'S WARRANTY	CATALOG DATA	OPERATING INSTRUCTI ONS	Design Data	OTHER	AP PROVED	DISAP PROVED	SEE REVERSE	INITIAL

BY COMPLET NG THIS FORM, THE UNDERSIGNED CONTRACTOR CERT FIES THAT THE MATERIAL COMPLIES OR IS COMPATIBLE WITH ALL SPECIFICATIONS OF THE SUBJECT CONTRACT

DATE	TYPE OR PRINT NAME AND TITLE		SIGNATURE:

TO ***(BASE CIVIL ENGINEERING OFFICER)***

For Evaluation and Action

DATE	TYPE OR PRINT NAME AND GRADE		SIGNATURE:

TO ***(AF Contracting Officer)***

RECOMMEND APPROVAL DISAPPROVAL AS INDICATED ABOVE AND SUBJECT TO ANY APPLICABLE COMMENTS ON THE REVERSE

DATE	TYPE OR PRINT NAME AND GRADE		SIGNATURE:

TO ***(Contractor)***

APPROVED DISAPPROVED AS INDICATED ABOVE AND SUBJECT TO ANY APPLICABLE COMMENTS ON THE REVERSE SIDE.

REQUEST RESUBMITTAL ON DISAPPROVED ITEMS WITHIN DAYS OF DATE SHOWN BELOW.

DATE	TYPE OR PRINT NAME AND GRADE		SIGNATURE:

COMMENTS

(Number to correspond with applicable Item Number on Reverse Side)

INSTRUCTIONS TO CONTRACTORS

1) *The term "material" is defined as articles, supplies, raw materials, equipment, parts, components, and end items that are to be incorporated into the work required by the contract.*

2) *This form is to be used by contractors for submitting Shop Drawings, Equipment Data, Manufacturer s Literature and Certificates and Samples of Materials to the Government for approval in accordance wit the provisions of this contract. Unless otherwise*

3) *Item (s) to be approved will be clearly tabbed or identified. Data pertaining to item (s) to be approved will be clearly identified or tabbed, particularly where documents are voluminous, in order to properly evaluate the materials or articles to be i*

4) *Requests submitted shall be numbered consecutively, by contract, in the space entitled "submission number". This number, in addition to the Contract No., will be used to identify each Material Approval Submittal. Resubmissions will be indicated in th*

5) *This Material Approval Submittal is not valid unless it is signed by the contracting officer. This approval is required as called for by the contracting officer under the terms of this contract.*

CAPE

TRANSMITTAL OF SHOP DRAWINGS, EQUIPMENT DATA, MATERIAL SAMPLES, OR MANUFACTURER'S CERTIFICATES OF COMPLIANCE *(Read instructions on the reverse side prior to initiating this form)*	DATE	TRANSMITTAL NO

SECTION I - REQUEST FOR APPROVAL OF THE FOLLOWING ITEMS *(This section will be initiated by the Contractor)*

TO:	FROM:	CONTRACT NO.	CHECK ONE: ☐THIS IS A NEW TRANSMITTAL ☐THIS IS A RESUBMITTAL OF TRANSMITTAL No.
SPECIFICATION SEC. NO. (Cover only one section with each transmittal)	PROJECT TITLE AND LOCATION		CHECK ONE: THIS TRANSMITTAL IS FOR ☐ FIO ☐ GOV'T APPROVAL

ITEM NO.	DESCRIPTION OF ITEM SUBMITTED *(Type size, model number, etc.)*	MFG OR CONTR. CAT., CURVE DRAWING OR BROCHURE NO. *(See instruction no. 8)*	NO. OF COPIES	CONTRACT REFERENCE DOCUMENT		FOR CONTRACTOR USE CODE	VARIATION *(See Instruction No. 6)*	FOR CE USE CODE
				SPEC. PARA. NO.	DRAWING SHEET NO.			
a.	*b.*	*c.*	*d.*	*e.*	*f.*	*g.*	*h.*	*I.*

REMARKS:

I certify that the above submitted items have been reviewed and are correct and in strict conformance with the contract drawings and specifications except as otherwise attached.

NAME AND SIGNATURE OF CONTRACTOR

SECTION II - APPROVAL AND ACTION

ENCLOSURES RETURNED (List by Item No.)	**NAME, TITLE AND SIGNATURE OF APPROVING AUTHORITY**	**DATE**

INSTRUCTIONS

1. Section I will be initiated by the Contractor in the required number of copies.

2. Each transmittal shall be numbered consecutively in the space provided for “Transmittal No.” This number, in addition to the contract number, will form a serial number for identifying each submittal. For new submittals or resubmittals mark the appropriate box; on resubmittals, insert transmittal number of last submission as well as the new submittal number.

3. The “Item No.” will be the same “Item No.” as indicated on ENG FORM 4288 for each entry on this form.

4. Submittals requiring expeditious handling will be submitted on a separate form.

5. Separate transmittal form will be used for submittals under separate sections of the specifications.

6. A check shall be placed in the “Variation” column when a submittal is not in accordance with the plans and specifications -- also, a written statement to that effect shall be included in the space provided for “Remarks.”

7. Form is self-transmittal, letter of transmittal is not required.

8. When a sample of material or Manufacturer’s Certificate of Compliance is transmitted, indicate “Sample” or “Certificate” in column c, Section I.

9. Army Corps of Engineers approving authority will assign action codes as indicated below in space provided Section I, column I to each item submitted. In addition, they will ensure enclosures are indicated and attached to the form prior to return to the contractor. The Contractor will assign action codes as indicated below in Section I, column g, to each item submitted.

THE FOLLOWING ACTION CODES ARE GIVEN TO ITEMS SUBMITTED

A -- Approved as submitted.	E -- Disapproved (See attached)
B -- Approved, except as noted on drawings.	F -- Receipt acknowledged
C - - Approved, except as noted on drawings. Refer to attached sheet resubmission required.	FX -- Receipt acknowledged, does not comply as noted with contract requirements.
D -- Will be returned by separate correspondence.	G -- Other *(Specify)*

10: Approval of items does not relieve the contractor from complying with all the requirements of the contract plans and specifications.

Reverse of ENG Form 4025

SUBMITTAL REGISTER

CONTRACT NUMBER

(ER 415-10)

TITLE AND LOCATION:

CONTRACTOR: Cape Environmental Management Inc

SPECIFICATION SECTION

ACTIVITY NO	TRANSMITTAL NO	ITEM NO	SPECIFICATION PARA NUMBER	DESCRIPTION OF ITEMS SUBMITTED	TYPE OF SUBMITTAL										CLASSIFICATION		REVIEWER	CONTRACTOR SCHEDULE DATE			CONTRACTOR ACTION			GOVERNMENT ACTION		REMARKS
					DATA	DRAWINGS	INSTRUCTIONS	SCHEDULE	STATEMENTS	REPORTS	CERTIFICATIONS	SAMPLES	RECORDS	O & M MANUAL	INFORMATION ONLY	GOVERNMENTS APPROVED		SUBMIT	APPROVAL NEEDED BY	MATERIAL NEEDED BY	CODE	DATE	SUBMIT TO GOVERN-MENT	CODE	DATE	
a	b	c	d	e	f	g	h	I	j	k	l	m	n	o	p	q	r	s	t	u	v	w	x	y	z	aa

ENG FORM 4288-R, JAN 97

EDITION OF MAR 95 IS OBSOLETE

(PROPONENT CEM--CE)

RE=RESIDENT ENG NEER ED=ENG NEERING DIVISION

SUBMITTAL REGISTER

CONTRACT NUMBER

(ER 415-10)

TITLE AND LOCATION:

CONTRACTOR: Cape Environmental Management Inc

SPECIFICATION SECTION

ACTIVITY NO	TRANSMITTAL NO	ITEM NO	SPECIFICATION PARA NUMBER	DESCRIPTION OF ITEMS SUBMITTED	TYPE OF SUBMITTAL										CLASSIFICATION		REVIEWER	CONTRACTOR SCHEDULE DATE			CONTRACTOR ACTION			GOVERNMENT ACTION		REMARKS
					DATA	DRAWINGS	INSTRUCTIONS	SCHEDULE	STATEMENTS	REPORTS	CERTIFICATIONS	SAMPLES	RECORDS	O & M MANUAL	INFORMATION ONLY	GOVERNMENTS APPROVED		SUBMIT	APPROVAL NEEDED BY	MATERIAL NEEDED BY	CODE	DATE	SUBMIT TO GOVERN-MENT	CODE	DATE	
a	b	c	d	e	f	g	h	I	j	k	l	m	n	o	p	q	r	s	t	u	v	w	x	y	z	aa

ENG FORM 4288-R, JAN 97

EDITION OF MAR 95 IS OBSOLETE

(PROPONENT CEM--CE)

RE=RESIDENT ENG NEER ED=ENG NEERING DIVISION

Appendix G: Final Sampling and Analysis Plan for Quantifying Total Petroleum Hydrocarbons Mass Holding Tank and Leach Tank Removal Action

Naval Facilities Engineering Systems Command Hawaii

Final Sampling and Analysis Plan for Quantifying Total Petroleum Hydrocarbons Mass Holding Tank and Leach Tank Removal Action

November 2021 Release, U.S. Navy Well [redacted]
JBPHH, O‘ahu, Hawai‘i

August 24, 2022

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Naval Facilities Engineering Systems Command Hawaii

Final Sampling and Analysis Plan for Quantifying Total Petroleum Hydrocarbons Mass Holding Tank and Leach Tank Removal Action

November 2021 Release, U.S. Navy Well [redacted]
JBPHH, O‘ahu, Hawai‘i

August 24, 2022

Prepared for NAVFAC Hawaii by

AECOM Technical Services Inc
1001 Bishop Street Suite 1600
Honolulu HI 96813-3698

N62742-17-D-1800
CTO N6274222F0106

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Table of Contents

Appendices

Tables

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Acronyms and Abbreviations

AECOM	AECOM Technical Services, Inc.
CAPE	Cape Environmental Management, Inc.
cm^3	cubic centimeter
CoC	chain of custody
CTO	contract task order
CY	cubic yard
DU	decision unit
EPA	Environmental Protection Agency, United States
g/cm^3	gram per cubic centimeter
ID	identification
JP-5	Jet Propellant 5
MI	multi-increment
MIS	multi-increment sampling
Navy	Department of the Navy, United States
PCS	petroleum-contaminated soil
POC	point of contact
RSD	relative standard deviation
SAP	sampling and analysis plan
TPH	total petroleum hydrocarbons
TPH-d	total petroleum hydrocarbons – diesel range organics
TPH-g	total petroleum hydrocarbons – gasoline range organics
TPH-o	total petroleum hydrocarbons – residual range organics (i.e., TPH-oil)

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1.0 Introduction and Purpose

This sampling and analysis plan (SAP) is provided to document the sampling and analytical requirements to quantify the mass of petroleum hydrocarbons removed from the Holding Tank and Leach Tank area of concern during a removal action done by others.

The purpose of this sampling and analysis is to provide defensible laboratory results for total petroleum hydrocarbons (TPH) in the petroleum-contaminated soil (PCS) that is removed during the removal action conducted by Cape Environmental Management, Inc. (CAPE). The removal action will be conducted by CAPE for the United States Department of the Navy (Navy) under a different contract. This plan is being provided under the Comprehensive Long-Term Environmental Action Navy contract N62742-17-D-1800, Contract Task Order (CTO) N6274222F0106.

This plan includes the following appendices:

- Appendix A – Project Schedule
- Appendix B – Analytical Data Package Requirements for Chemical Analyses
- Appendix C – Attachment 1. Sample Log Form

2.0 Background

On November 20, 2021, a release of Jet Propellant 5 (JP-5) jet fuel occurred in the Adit 3 tunnel of the Red Hill Bulk Fuel Storage Facility (Facility). JP-5 was released from an overhead 14-inch polyvinyl chloride pipe at a location [redacted]

Much of that JP-5 accumulated in the Adit 3 sump and migrated within the sump drainage system via a sump pump to approximately [redacted] from the entrance of Adit 3. JP-5 migrated another [redacted] under the Adit 3 loading area to reach the sump drain holding tank and connected sump drain leach tank located at the northwest perimeter of the Facility, adjacent to South Hālawa Stream. Following an initial and Phase 2 site characterization of the subsurface soil and perched water in the area surrounding the holding tank and leach tank conducted by the Navy, an initial removal action was conducted by the Navy to remove the holding tank and leach tank infrastructure and limited PCS.

This SAP addresses a second removal action conducted by the Navy, which will remove an additional 1,000 cubic yards (CY) of soil from the site and is documented in the removal action plan (DON 2022).

3.0 Regulatory Framework

This SAP has been developed to meet Navy requirements to estimate the amount of fuel that has been treated or removed during the removal action operations. This SAP does not address regulatory requirements, and assumes that sampling and analysis to address specific regulatory requirements are described in a different document.

4.0 Roles and Responsibilities

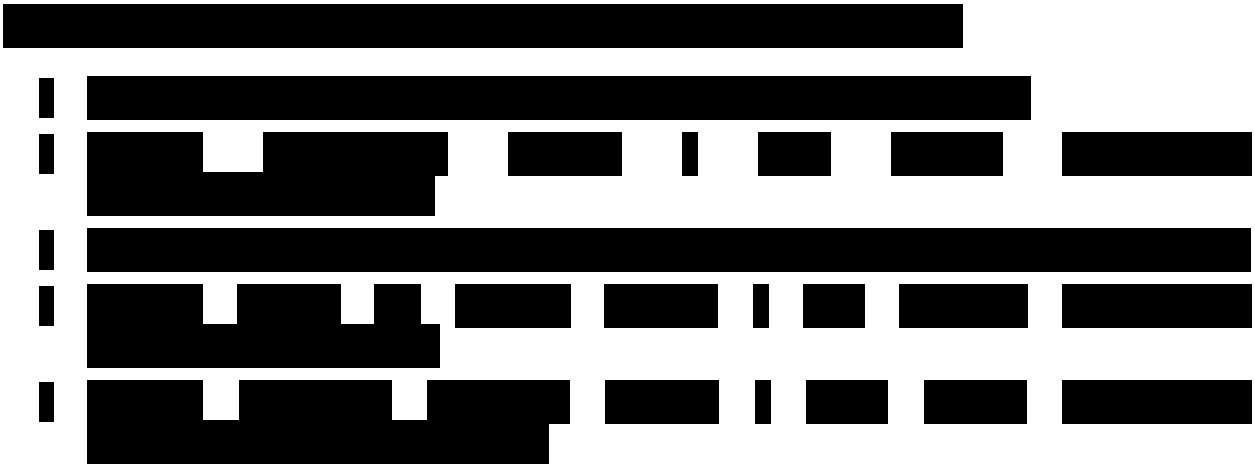
This SAP has been developed to document sampling and analysis procedures that will be conducted by the Navy, including prime Navy contractors under two separate contracts, as discussed in the following sections.

4.1 Navy TPH Quantification Contractor

AECOM Technical Services, Inc. (AECOM) is the Navy TPH Quantification Contractor, supporting this task in accordance with N62742-17-D-1800, CTO N6274222F0106 to:

1) Develop this SAP.
2) Subcontract and manage the project analytical laboratory Subcontractor.
3) Provide sampling materials to the Navy Sampling Contractor.
4) Collect properly containerized samples from the Navy Sampling Contractor, and pack and ship those samples to the project analytical laboratory Subcontractor for analysis.
5) Evaluate the results from the project analytical laboratory Subcontractor.

4.1.1 Navy TPH Quantification Contractor Contact Information



4.2 Navy Sampling Contractor

CAPE is the Navy Sampling Contractor, supporting this task in accordance with N6274216D1807, CTO N6274222F0135 to:

- Work closely with the Navy TPH Quantification Contractor to:
  - Provide input to ensure this SAP can be implemented in an efficient and safe manner.
  - Communicate expected schedule and revised schedules as they change.
  - Provide documentation necessary to complete the TPH quantification.

- Collect sampling material provided by the Navy TPH Quantification Contractor at the agreed upon location and times to meet the requirements of this SAP.
- Collect, label, containerize, preserve, store, and document samples associated with this SAP during the removal action in a safe and efficient manner and in accordance with this SAP.
- Provide properly stored samples and documentation to the Navy TPH Quantification Contractor at times and dates that are mutually agreed upon by all parties.
- Provide additional information to the Navy TPH Quantification Contractor, including:
  - Completed chain-of-custody (CoC) forms
  - Completed logs of samples related to associated load disposal manifests with load weights
  - Other items identified during the planning and implementation of the sampling and analysis associated with this SAP

4.2.1 Navy Sampling Contractor Contact Information

[redacted]

5.0 Data Quality Objectives

The data quality objectives for the near-surface intrusive assessments are described in this section.

Conduct scientifically defensible quantitative sampling and analysis of soil removed during the holding tank and leach tank removal action to calculate a mass of petroleum hydrocarbon removed in the carbon range from C6 to C40. This hydrocarbon carbon range encompasses the mixture of compounds that make up JP-5, which is a middle distillate, similar to kerosene.

To meet this objective, the following analytical methods will be used to evaluate this carbon range:

- TPH in the gasoline range (TPH-g, C6 to C10) by United States Environmental Protection Agency (EPA) Method 8260D/CALUFT
- TPH in the diesel range (TPH-d, C10-C24), by EPA 8015C Modified
- TPH in the residual oil range (TPH-o, C24-C40) by EPA 8015C Modified

It is estimated that approximately 1,200 CY of fluffed PCS will be removed for disposal. Multi-increment (MI) sampling (MIS) methodology will be used to estimate the average concentration of the TPH fractions within each decision unit (DU). To achieve adequate precision in estimating average TPH concentrations, 30 increments will be incorporated into the MI sample for each DU. Precision will be evaluated by collecting replicate MI samples (triplicates) for two DUs and evaluated for precision as described in Section 6.2.3. Samples will be collected as follows:

- For this project, a DU will be approximately 90 CY of PCS.
  - 1,200 CY-PCS / 90 CY-PCS/DU = approximately 13 DUs.
- Each DU will be represented by a single MI sample each consisting of 30 increments.
  - An increment will be collected at approximately every 3 CY of PCS removed to the landfill (approximately one increment for every excavator bucket).
  - Each truck will contain approximately 15 CY.
  - One 30-increment MI sample will be collected for approximately every 90 CY removed.
- For simplification, the following sampling scheme will be conducted:
  - 5 increments per truck × 6 trucks = 30 increments per MI sample.
- Each MI increment will be collected from the center of the backhoe bucket and placed in the MIS container for the DU.
- Triplicate samples will be collected from two DUs for the evaluation of variability. Increments for triplicate samples will be collected from different locations in the backhoe buckets.

To calculate a mass of TPH removed from the MI sample concentrations measured by the laboratory, the mass of the entire DU must be recorded and used in the calculation:

$$M_{TPH}(mg) = C_{TPH}(mg/kg) \times M_{soilDU}(kg)$$

Therefore, each truck associated with a given MI sample must be tracked, and each truckload must be weighed for soil mass. Each truckload will be represented by a load manifest document number, and the load manifest number must be recorded on the CoC form for each MI sample collected. At the landfill, the manifest will be completed with the load weight and provided back to the Navy to document the weight of the soil associated with each MI sample. Each MI sample and associated truck manifests will be documented in the field using the Tracking Log Form provided as Attachment 1 (Appendix C). Based on the simplified sampling scheme described above, each DU will be associated with six (6) truckload manifests, which will be noted on the DU log form.

6.0 Holding Tank and Leach Tank Removal Action TPH Quantification Sampling Strategy

6.1 Pre-sampling Planning and Coordination

Following the authorization to proceed by the Navy Contracting Officer, and prior to development of the SAP, the Navy will conduct an initial kick-off coordination meeting. This meeting will include the Navy TPH Quantification Contractor and the Navy Sampling Contractor to document the general project requirements and to introduce the POCs from the Navy and prime contractors, and to identify a schedule of deliverables.

Following the kick-off meeting, the Navy TPH Quantification Contractor will develop this SAP and, in conjunction, identify and subcontract a Department of Defense-certified analytical laboratory that can perform the MIS analytical protocols to meet the data quality objectives as described in Section 5.0. The analytical laboratory will complete tables in Appendix B and provide tables including their project-specific detection limits, sample quantities, containers, preservatives, hold times, and other specifics.

The Navy TPH Quantification Contractor will provide this draft SAP to the Navy, who will review and provide comments. It is anticipated that the Navy will provide the draft SAP to the Navy Sampling Contractor for their comments and for inclusion in their Draft Removal Action SAP as an attachment or appendix based on the schedule provided in Appendix A.

Following submittal of Navy and Navy Sampling Contractor comments on the SAP, the Navy will conduct a pre-sampling coordination meeting to answer any questions the Navy TPH Quantification Contractor has before finalizing the SAP.

Once the SAP has been finalized, the Navy Quantification Contractor will order sample supplies from the project laboratory and prepare sampling kits for the Navy Sampling Contractor.

6.2 TPH Quantification Sampling

The Navy TPH Quantification Contactor will provide at least half of the required sampling kits to the Navy Sampling Contractor no later than 3 days before the first day of the initial PCS excavation and disposal activities to allow the Navy Sampling Contractor to prepare labels and COCs prior to the removal action sampling events.

PCS is defined as subsurface soil deeper than 8 feet below ground surface and within the PCS footprint provided by the TPH Quantification Subcontractor. Therefore, soil that is above 8 feet below ground surface and/or is not within the excavation footprint should not be included in the contracted 1,000 CY of PCS to be removed, and should not be sampled for this quantification of TPH mass.

6.2.1 Multi-Increment Sampling Methodology

As described in Section 5.0, TPH quantification sampling will be accomplished using MIS methodology.

Each MI sample or replicate sample will be collected as follows:

- Each sample will consist of 30 increments of soil.
- A 30-increment aliquot for TPH-d and TPH-o will consist of approximately 1 kilogram of soil.
- Each soil increment will be collected with a sample-specific, disposable, and certified-clean scoop that will be disposed of after each MI sample is collected.
  - Each soil increment will be approximately 1/30 kilogram or, assuming a loose soil density of 1.66 grams per cubic centimeter (g/cm^3), 33.33 g/1.66 g/cm^3 = approximately 20.83 cm^3 per scoop.
- A 30-increment aliquot for TPH-g will consist of 30, 5-cm^3 increments of soil:
  - Each 5-cm^3 soil increment will be collected with a sample-specific, disposable, and certified-clean Terra-Core T-Sample, that will be disposed of after each MI sample is collected.
  - TPH-g increments will be preserved in methanol.
  - Each MI sample for the TPH-g analysis will be containerized in six pre-tared, 4-ounce jars, each with 25 cm^3 of methanol and Teflon caps.
  - Five soil increments will be containerized in each 4-ounce jar so that 25 cm^3 of soil will be preserved in 25 cm^3 of methanol.
  - Weights of tared containers must not be altered by the addition of labels or other matter that is not the sample material.
  - Each 4-ounce jar will be provided with dedicated bubble wrap that must be replaced around the container before packed together with the entire TPH-g sample set in a 1-gallon Ziploc plastic bag.
  - A completed sample label should be placed on the outside of the inner Ziploc bag, then the inner Ziploc bag should be evacuated of air, sealed, then placed in a second Ziploc bag, which will be sealed and taped closed such that the inner bag label can be clearly read during final sample packing and shipping and at the laboratory without opening the taped outer bag. This is important for quality assurance to ensure the COCs are correct throughout the process.

6.2.2 Coordination Removal Action Activities With TPH Quantification Sampling

The Navy Sampling Contractor is responsible for ensuring that all sampling is conducted in a safe manner and must develop a Task Hazard Analysis that clearly describes the steps that will be conducted to perform the sampling safely.

As described in Section 5.0, this SAP assumes the following:

- Approximately 1,200 CY of fluffed PCS will be included in the TPH quantification analysis.
- Soil will be loaded into each truck in excavator buckets that will hold approximately 3 CY of loose soil.
- Each truck will carry approximately 15 CY of loose soil.
- As an approximation, 1 sample increment (1 scoop for TPH-d and TPH-o; 1 5 cm^3 T-sample plug for TPH-g) should be collected from every excavator bucket of soil transferred from the excavation to the truck.
- Samples must have a minimum of 30 increments per sample.
- The increments will be associated with six trucks (5 increments per truck × 6 trucks = 30 increments per MI sample).

To provide flexibility and safety, aliquots of soil from each bucket can be placed on a Visqueen-lined surface during the active loading process (five per truck), and these aliquots can be sampled after active loading is completed. However, it is imperative that these increments are properly associated with the specific truck manifest on the MI sample log sheet.

6.2.3 Quality Assurance Replicate Samples

Two DUs will have replicate samples collected to estimate the variability associated with the incremental samples for the DU.

One set of three replicates will be collected in the northeast portion of the removal action in the vicinity of the holding tank (DU01) and a second set of three replicates will be collected in the northeast portion of the removal action in the vicinity of the leach tank (DU07).

Replicates are MI samples that are collected simultaneously from the same excavator buckets and can be collected with the same dedicated scoop and 5 cm^3 T-sampler but in three different soil sampling kit sets.

For example, for DU1, for each sampled excavator bucket:

- The DU01-R1 soil increment may be collected from the right upper corner of the bucket.
- The DU01-R2 soil increment may be collected from the center of the bucket.
- The DU01-R3 soil increment may be collected from the right upper corner of the bucket.

A similar consideration should be taken if soil increments are collected from bucket aliquots set on Visqueen as described as an option in Section 6.2.2.

Replicate samples will be collected in exactly the same way as the normal MI sample, just from different locations within the DU.

Replicate data precision will be evaluated by calculating the relative standard deviation (RSD) of TPH concentrations for each of the two DUs. The RSD represents the precision of field and laboratory errors combined. Lower RSDs indicate higher levels of precision, reproducibility, and reliability of the data.

7.0 Sample Details

Details of subsurface unconsolidated material samples are presented in Appendix B.

7.1 Sample Custody Requirements

Each sample will be assigned a CoC sample identification (ID) number and a descriptive ID number in accordance with Naval Facilities Engineering Systems Command, Pacific, Environmental Restoration Program Procedure I-A-8, *Sample Naming* (DON 2015). All sample ID numbers will be recorded in the field logbook in accordance with Procedure III-D, *Logbooks* (DON 2015). The CoC sample ID number (the only ID number submitted to the analytical laboratory) is used to facilitate data tracking and storage. The CoC sample ID number allows all samples to be submitted to the laboratory without providing information on the sample type or source. The descriptive ID number is linked to the CoC sample ID number, which provides information regarding sample type, origin, and source.

7.1.1 CoC Sample Identification Number

A CoC sample ID number will be assigned to each sample as follows to facilitate data tracking and storage.

7.1.2 Descriptive Sample Identifier

A descriptive ID number (for internal use only) will identify the sampling location, type, sequence, matrix, and depth. The descriptive ID number is used to provide sample-specific information (e.g., location, sequence, and matrix). The descriptive identifier is not revealed to the analytical laboratory. The descriptive ID number for all samples is assigned as follows:

AAbb-Cd-eeffgg-hhiijj

Where:

AA = "DU" for Decision Unit (Table 7-1)

bb = Integer no. incremented chronologically based on DU. no. (e.g., 01, 02)

C = Sample Type ("M" for MIS, "R" for Replicate)

d = Integer no. incremented chronologically based on sample type per DU (e.g., 1, 2)

ee = Beginning numerical sample date year ("22" for 2022)

ff = Beginning numerical sample date month ("09" for September)

gg = Beginning numerical sample date day ("12" for day 12 of the month)

hh = Ending numerical sample date year ("22" for 2022)

ii = Ending numerical sample date month ("09" for September)

jj = Ending numerical sample date day ("13" for day 13 of the month)

For example, the sample number DU03-R3-220915-220916 would indicate that:

- The sample is from the third set of trucks (DU03).
- The third replicate of 3 collected from this DU03 (R3).
- The first increment was collected on September 15, 2022 (220915).
- The last increment was collected on September 16, 2022 (220915).

Table 7-1: Sample Identifiers

Identifier	Definition
DU#	DUs are specific truckloads associated with each MI or replicate sample, chronologically numbered and associated by log form.

Table 7-2: Sample Type and Matrix Identifiers

Identifier	Sample Type	Matrix
M#	MI Sample, # is always 1	Excavated earth (rock and soil)
R#	Replicate sample, # between 1 and 3	Excavated earth (rock and soil)

7.1.3 Handling, Shipping, and Custody

All samples collected for analysis will be recorded in the field logbook in accordance with Procedure III-D, *Logbooks* (DON 2015). All samples will be labeled and recorded on CoC forms in accordance with Procedure III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody* (DON 2015). Samples will be handled, stored, and shipped in accordance with Procedure III-F, *Sample Handling, Storage, and Shipping* (DON 2015). All samples collected on this project will be shipped to the analytical laboratory via overnight airfreight.

All samples received at the analytical laboratory will be managed in accordance with laboratory standard operating procedures for receiving samples, archiving data, and sample disposal and

waste collection, as well as storage and disposal per Section 5.8, "Handling of Samples" of the Department of Defense *Quality Systems Manual* v. 5.4 (DoD and DOE 2021).

7.2 Laboratory Quality Control Samples

Laboratory quality control samples will be prepared and analyzed in accordance with the methods and procedures listed in Appendix B.

8.0 References

Department of Defense and Department of Energy, United States (DoD and DOE). 2018. *Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories*. DoD QSM Ver. 5.1.1. Prepared by DoD Environmental Data Quality Workgroup and DOE Consolidated Audit Program Operations Team.

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Department of the Navy (DON). 2015. *Final Project Procedures Manual, U.S. Navy Environmental Restoration Program, NAVFAC Pacific*. JBPHH HI: Naval Facilities Engineering Command, Pacific. May.

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Technical Services, Inc., Honolulu, HI. Prepared for Defense Logistics Agency Energy, Fort Belvoir, VA, under Naval Facilities Engineering Systems Command, Hawaii, JBPHH HI.

———. 2021b. *Initial Release Response Report, Pipeline Breach in Tunnel Red Hill Bulk Fuel Storage Facility JBPHH O‘ahu Hawai‘i*. Prepared by AECOM Technical Services, Inc. JBPHH HI: Naval Facilities Engineering Systems Command, Hawaii. September.

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Appendix A – Project Schedule

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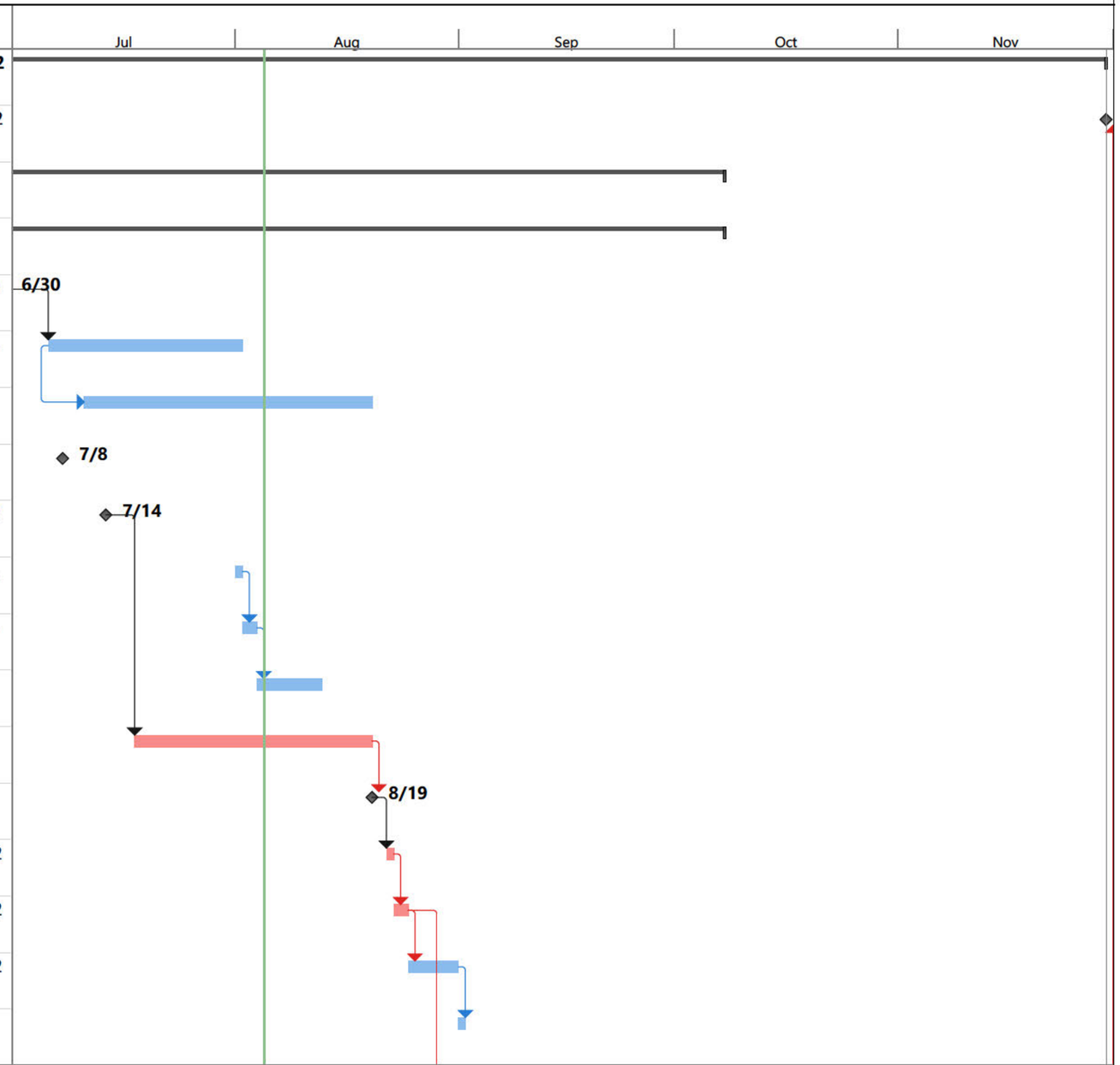
Project Schedule
Red Hill Concrete Tank Removal Project

DD: Thu 6/30/22

ID	Task Name	Duration	Start	Finish
1	**Red Hill Concrete Tank Removal, JBPHH, HI**	**109 days**	**Thu 6/30/22**	**Tue 11/29/22**
2	Project Complete	0 days	Tue 11/29/22	Tue 11/29/22
3	**WE 5 Options (Mod 01)**	**72 days**	**Thu 6/30/22**	**Fri 10/7/22**
4	**Task 22: Implement Option 1**	**72 days**	**Thu 6/30/22**	**Fri 10/7/22**
5	Award Mod 01 (Option 1)	0 days	Thu 6/30/22	Thu 6/30/22
6	Procure Engineering Subcontractor	19 days	Wed 7/6/22	Mon 8/1/22
7	Procure vendors (heavy equipment, trucking, T&D, etc)	30 days	Mon 7/11/22	Fri 8/19/22
8	1st Status Meeting with ACOR/FEAD	0 days	Fri 7/8/22	Fri 7/8/22
9	Site Visit with ACOR/FEAD/Engineering Sub	0 days	Thu 7/14/22	Thu 7/14/22
10	Survey Site	1 day	Mon 8/1/22	Mon 8/1/22
11	Receive Revised Topo	2 days	Tue 8/2/22	Wed 8/3/22
12	Shoring/Excavation Plan	7 days	Thu 8/4/22	Fri 8/12/22
13	Modify Work Plan/Safety Plan	25 days	Mon 7/18/22	Fri 8/19/22
14	Submit Draft Work Plan	0 days	Fri 8/19/22	Fri 8/19/22
15	Navy Review Work Plan	1 day	Mon 8/22/22	Mon 8/22/22
16	Revised Draft Work Plan	2 days	Tue 8/23/22	Wed 8/24/22
17	Regulator Review Draft Plans	5 days	Thu 8/25/22	Wed 8/31/22
18	Final Work Plan	1 day	Thu 9/1/22	Thu 9/1/22

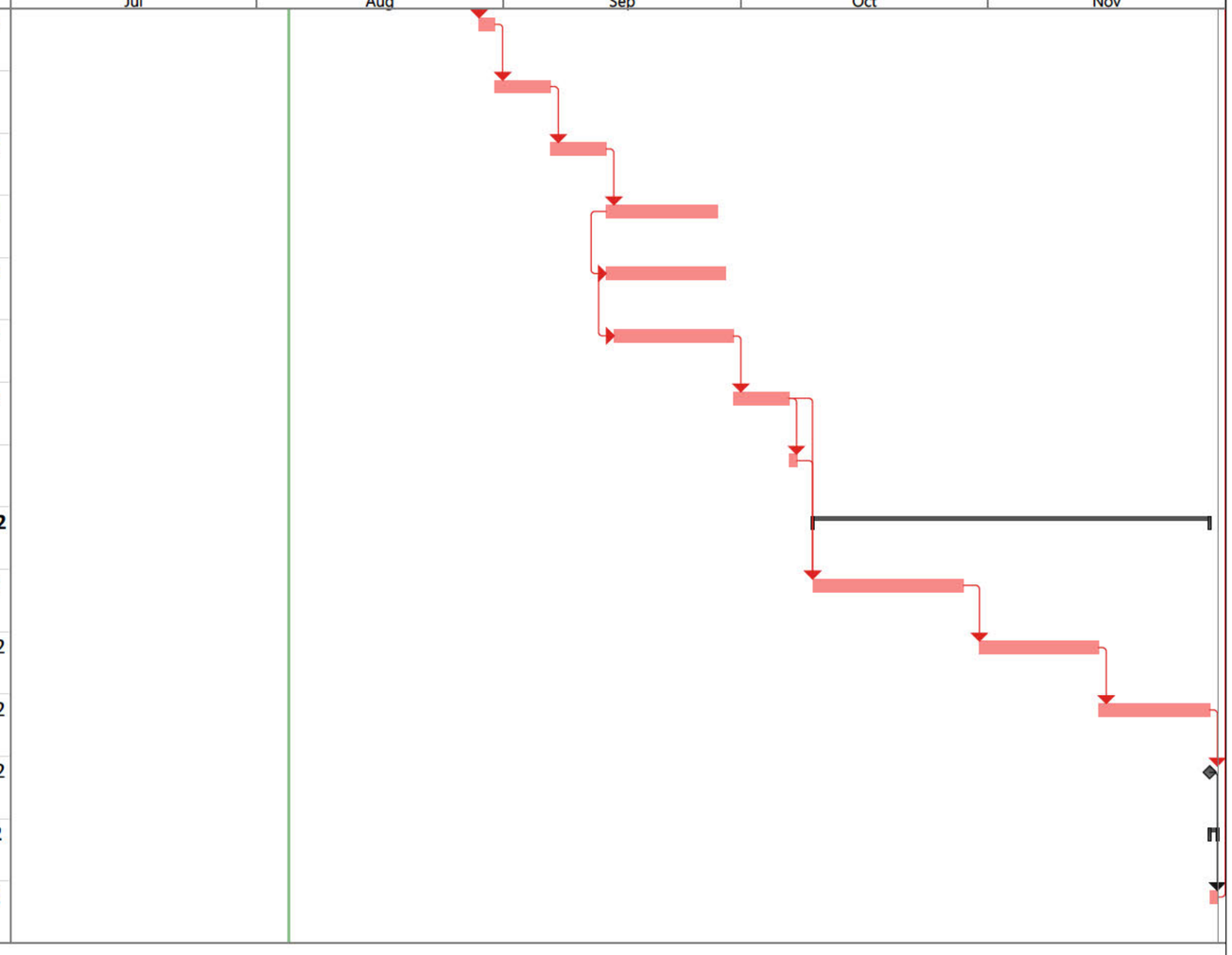


Task Milestone Summary Project Summary Critical Progress

Project Schedule
Red Hill Concrete Tank Removal Project

DD: Thu 6/30/22

ID	Task Name	Duration	Start	Finish
19	Mobilization	2 days	Mon 8/29/22	Tue 8/30/22
20	Site Setup	5 days	Wed 8/31/22	Tue 9/6/22
21	Excavate Overbuden and Layback Walls	5 days	Wed 9/7/22	Tue 9/13/22
22	Excavate up to 1,000 cyds and T&D to Landfill	10 days	Wed 9/14/22	Tue 9/27/22
23	Confirmation Sampling & Results	11 days	Wed 9/14/22	Wed 9/28/22
24	Backfill	11 days	Thu 9/15/22	Thu 9/29/22
25	Restoration and Replace Fence	5 days	Fri 9/30/22	Thu 10/6/22
26	Demobilization	1 day	Fri 10/7/22	Fri 10/7/22
27	**WE 6 Reporting**	**36 days**	**Mon 10/10/22**	**Mon 11/28/22**
28	Draft Closure Report	15 days	Mon 10/10/22	Fri 10/28/22
29	Navy Review	11 days	Mon 10/31/22	Mon 11/14/22
30	RTC	10 days	Tue 11/15/22	Mon 11/28/22
31	Final Closure Report	0 days	Mon 11/28/22	Mon 11/28/22
32	**WE 7 Closeout**	**1 day**	**Tue 11/29/22**	**Tue 11/29/22**
33	Closeout	1 day	Tue 11/29/22	Tue 11/29/22



Task | Milestone | Summary | Project Summary | Critical | Progress

Appendix B – Analytical Data Package Requirements for Chemical Analyses

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GC-FID Stage 2 Deliverables

Item No.	Deliverable
1	Chain of Custody
2	Sample results with analysis and extraction/preparation dates
3	Summary of MS/MSD/duplicate recoveries and control limits (listing or link with associated samples)
4	Summary of LCS/LCSD recoveries and control limits (listing or link with associated samples)
5	Method blanks (listing or link with associated samples)
6	Summary of surrogate recoveries (listing or with sample results)
7	Case narrative to discuss anomalies

Note: The data deliverable package must have a table of contents and be paginated.
GC-FID gas chromatography-flame ionization detector
MS matrix spike
MSD matrix spike duplicate
LCS laboratory control sample
LCSD laboratory control sample duplicate

GC-MS Stage 2 Deliverables

Item No.	Deliverable
1	Chain of custody
2	Sample results with analysis and extraction/preparation dates
3	Summary of MS/MSD/Duplicate recoveries and control limits (listing or link with associated samples)
4	Summary of LCS/LCSD recoveries and control limits (listing or link with associated samples)
5	Method blanks (listing or link with associated samples)
6	Summary of surrogate recoveries (listing or with sample results)
7	Case narrative to discuss anomalies

Note: The data deliverable package must have a table of contents and be paginated.
GC-MS gas chromatography-mass spectrometry

General Chemistry Stage 2 Deliverables

Item No.	Deliverable
1	Chain of custody
2	Sample results with analysis and extraction/preparation dates
3	Summary of MS/MSD/duplicate recoveries and control limits (listing or link with associated samples)
4	Summary of LCS/LCSD recoveries and control limits (listing or link with associated samples)
5	Method blanks (listing or link with associated samples)
6	Case narrative to discuss anomalies

Note: The data deliverable package must contain a table of contents and be paginated.

HARD COPY DATA DELIVERABLES COMPACT DISK REQUIREMENTS

The compact disc (CD), if requested, shall contain exactly the same information as the hard copy data deliverables including amended and additional pages requested during data review. Upon completion of data review by AECOM Technical Services, Inc. or a third party, the laboratory may be requested to provide the CD with the following:

- The images shall be clear and legible.
- The images shall be right side up.
- The images shall be straight.
- The images shall be in the same order as the hard copy data deliverables.
- Images may be submitted in Portable Document Format (PDF), Tag Image File Format (TIFF), or other equivalent imaging format. Files shall be burned for each page and each CD shall be indexed. The laboratory shall log in samples based on project number, project name, and sample delivery group (also known as batch or work order).
- If the images are not clear, legible, right side up, straight, or in order, then the laboratory shall resubmit the CD.
- The CD label shall contain the following information:
  - Navy contract number
  - CTO name and number
  - Sample delivery group number
  - Matrices and methods
  - Date of submittal

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Appendix B.2:

Field Sampling, Analytical, and
Quality Management Reference Tables

- Table B-1: Location-Specific Sampling Methods/SOP Requirements
- Table B-2: Analyte List and Reference Limits
- Table B-3: Preparation and Analytical Requirements for Field and QC Samples
- Table B-4: Analytical Services
- Table B-5: Analytical SOP References
- Table B-6: Laboratory QC Samples
- Table B-7: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
- Table B-8: Analytical Instrument Calibration
- Table B-9: Data Verification and Validation (Steps I and IIa/IIb) Process

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APPENDIX B.2 – ACRONYMS AND ABBREVIATIONS

%	percent
CCV	continued calibration verification
DoD	Department of Defense
EICP	extracted ion current profile
EPA	Environmental Protection Agency, United States
GC	gas chromatography
GC-FID	gas chromatography-flame ionization detector
GC-MS	gas chromatography-mass spectrometry
ICAL	initial calibration
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
MS	matrix spike
MSD	matrix spike duplicate
QA	quality assurance
QC	quality control
QSM	Quality Systems Manual
RT	retention time
SOP	standard operating procedure
TBD	to be determined

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Table B-1: Location-Specific Sampling Methods/SOP Requirements

Sampling Location/ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Sampling SOP Reference
DU1-M1-BegDate-EndDate DU2-M1-BegDate-EndDate DU3-M1-BegDate-EndDate DU4-M1-BegDate-EndDate DU5-M1-BegDate-EndDate DU6-M1-BegDate-EndDate DU7-M1-BegDate-EndDate DU8-M1-BegDate-EndDate DU9-M1-BegDate-EndDate DU10-M1-BegDate-EndDate DU11-M1-BegDate-EndDate DU12-M1-BegDate-EndDate DU13-M1-BegDate-EndDate DU1-R1- BegDate-EndDate DU1-R2- BegDate-EndDate DU1-R3- BegDate-EndDate DU5-R1- BegDate-EndDate DU5-R2- BegDate-EndDate DU5-R3- BegDate-EndDate	Unconsolidated Material	8 to 20	TPH-g, 8260/CALUFT TPH-d, 8015Mod TPH-o, 8015Mod	MI Samples: • 1 increment every 3CY • 30 increments per MI sample REP Samples: • 1 increment every 3CY per Rep#1, Per Rep#2 • 30 increments per MI sample	Procedure I-B-1, *Soil Sampling*

Notes:
Procedures are from the *Project Procedures Manual* (DON 2015).
Actual number of unconsolidated material samples will be dependent on field observations during coring.
One trip blank will be collected during each unconsolidated material sampling event.
TPH with silica gel cleanup will only be analyzed for samples with detections of TPH-d and TPH-o from the non-silica gel cleaned extract.
bgs below ground surface
SOP standard operating procedure

Table B-2: Location-Specific Sampling Methods/SOP Requirements

Matrix Unconsolidated Material

Analyte	CAS Number	Screening Criterion [a] (mg/kg)	Project LOQ Goal (mg/kg)	Project LOD Goal (mg/kg)	Laboratory-Specific Limits (mg/kg)		
					LOQ	LOD	DL
TPH							
TPH-g (C6–C10)	N/A	450/100/700 [b]	33	10	4.0	3.0	1.3
TPH-d (C10–C24)	N/A	220/500/940	73	22	50	30	9.9
TPH-o (C24–C40)	N/A	9,400/500/1,000	150	50	50	30	20

CAS Chemical Abstracts Service
DL detection limit
LOD limit of detection
LOQ limit of quantitation
mg/kg milligram per kilogram
N/A not applicable
[a] State of Hawai‘i Department of Health Tier 1 EALs (summer 2016, updated January 2017).
[b] EALs for direct exposure/gross contamination/leaching to groundwater.

Table B-3: Preparation and Analytical Requirements for Field and QC Samples

Matrix	Analytical Group	Preparation Reference/Method SOP Analytical Reference/Method SOP	Containers	Sample Volume	Preservation Requirement	Maximum Holding Time (preparation/analysis)
MIS Unconsolidated Material	TPH-g	Preparation: EPA 5035C/CA-T-WI-012 Analysis: EPA 8260/CALUFT / EFGS-T-VOA-SOP41119	6 × 4-oz glass jar	5 × 5 g per jar; 6 jars per sample (total 30 5-g increments)	25 mL methanol per jar; cool ≤6°C	N/A/14 days
	TPH-d, TPH-o	MIS Preparation: SOP EFGS-T-OP-SOP41256 Extraction: EPA 3546/EFGS-T-OP-SOP41432 v1 Analysis: EPA 8015D/EFGS-T-GCS-SOP40900 v1	1-gallon Ziploc bag	1 kg (30 increments, 30-40 g each)	Cool ≤6°C	14 days/40 days

°C degree Celsius
g gram
kg kilogram
mL milliliter
N/A not applicable
oz ounce

Table B-4: Analytical Services

Matrix	Analytical Group	Sampling Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (name and address)
MIS Unconsolidated Material	TPH-g	DU1-M1-BegDate-EndDate Thru DU13-M1-BegDate-EndDate DU1-R1- BegDate-EndDate DU1-R2- BegDate-EndDate DU1-R3- BegDate-EndDate DU5-R1- BegDate-EndDate DU5-R2- BegDate-EndDate DU5-R3- BegDate-EndDate	EFGS-T-VOA-SOP41119	14 days after samples are received at laboratory	Eurofins Environment Testing Northwest, LLC (Eurofins Seattle) 5755 8th Street East Tacoma, WA 98424
MIS Unconsolidated Material	TPH-d TPH-o	DU1-M1-BegDate-EndDate Thru DU13-M1-BegDate-EndDate DU1-R1- BegDate-EndDate DU1-R2- BegDate-EndDate DU1-R3- BegDate-EndDate DU5-R1- BegDate-EndDate DU5-R2- BegDate-EndDate DU5-R3- BegDate-EndDate	EFGS-T-GCS-SOP40900 v1	14 days after samples are received at laboratory	Eurofins Environment Testing Northwest, LLC (Eurofins Seattle) 5755 8th Street East Tacoma, WA 98424

[a] The laboratory meets DoD Environmental Laboratory Accreditation Program or American Association of State Highway and Transportation Officials accreditation requirements, as applicable, to support project needs.

Table B-5: Analytical SOP References

Laboratory: TBD
Point of Contact: TBD
Point of Contact Phone Number: TBD

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM (Yes/No)	Modified for Project Work? (Yes/No)
Preparatory Methods						
EFGS-T-OP-SOP41256	Incremental Subsampling Method of Soils and Sediments	Definitive	TPH-d, TPH-o (MIS Unconsolidated Material)	Preparation	No	No
CA-T-WI-012	ISM Options for VOCs	Definitive	TPH-g (MIS Unconsolidated Material)	Preparation	No	No
EFGS-T-OP-SOP41432 v1	Microwave Extraction Procedure [EPA Method 3546]	Definitive	TPH-d, TPH-o, PAHs (MIS Unconsolidated Material)	Preparation	No	No
Analytical Methods						
EFGS-T-MSS-SOP41389 v1	Volatile Organic Compound Analysis by GC/MS [Method 8260D]	Definitive	TPH-g (MIS Unconsolidated Material)	GC-MS	No	No
EFGS-T-GCS-SOP40900 v1	Extractable Petroleum Fuel Hydrocarbons [Method 8015B and D Mod]	Definitive	TPH-d, TPH-o (MIS Unconsolidated Material, Water)	GC-FID	No	No

Note: The laboratory SOPs listed in the table are the most current revisions at the time of publication of this sampling and analysis plan. The Navy consultant will review the laboratory SOPs immediately prior to sample submittal to ensure that the laboratory uses SOPs that are in compliance with the DoD QSM annual review requirement.

GC-FID gas chromatography-flame ionization detector
GC-MS gas chromatography-mass spectrometry
PAH polynuclear aromatic hydrocarbon
QSM Quality Systems Manual
VOC volatile organic compound

Table B-6: Laboratory QC Samples for Chemistry Analyses

Matrix	**Unconsolidated Material (MIS)**
Analytical Group	**TPH-g**
Analytical Method/SOP Reference	Analytical Method: SW-846 8260C/CALUFT Preparation Method: EPA 5035A, MIS Laboratory SOPs: CA-T-WI-012, EFGS-T-VOA-SOP41119
Analytical Organization	Eurofins Seattle

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.4 (DoD and DOE 2021).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify the method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP ANA8260 and at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).
Tune check	Prior to the ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No samples may be analyzed without a passing tune.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±20% of true value. All reported analytes and surrogates within ±50% for the end of the analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate, then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. If the specific version of a method requires additional evaluation (e.g., average response factors), these additional requirements must also be met.
MB	Each time analytical batch.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per DoD QSM Appendix C Limits, Method SW-846 8260C and Lab SOP.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes. Results may not be reported without a valid LCS.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per DoD QSM Appendix C Limits, Method SW-846 8260C and Lab SOP. MSD or Matrix Duplicate: RPD of all analytes ≤20%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ±10 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision/ Representativeness	Laboratory in-house method manual to be followed for acceptance criteria.
Surrogate spike	All field and QC samples.	Per DoD QSM Appendix C Limits, Method SW-846 8260C, and Lab SOP.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).
Trip blank	1 per cooler.	Target analytes ≤1/2 LOQ.	Reanalyze for confirmation through a second analysis of the trip blank. Examine the PQOs.	Analyst Lab QA Officer Project Chemist	Accuracy/Bias, Representativeness/ Contamination	Target analytes ≤1/2 LOQ.

%	percent
BFB	4-bromofluorobenzene
CCV	continued calibration verification
DFTPP	decafluorotriphenylphosphine
DQI	data quality indicator
EICP	extracted ion current profile
ICAL	initial calibration
MB	method blank
PQO	project quality objective
QA	quality assurance
RPD	relative percent difference

Matrix **Unconsolidated Material**
Analytical Group **TPH-d, TPH-o without Silica Gel Cleanup**
Analytical Method/SOP Reference Analytical Method: EPA Method 8015C
Preparation Method: MIS prep, EPA 3550C
Laboratory SOPs: EFGS-T-OP-SOP41256, EFGS-T-OP-SOP41432 v1, EFGS-T-GCS-SOP40900 v1
Analytical Organization Eurofins Seattle

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representative-ness	QC acceptance criteria as specified by Lab SOP.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.4 (DoD and DOE 2021).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP, and at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per DoD QSM Appendix C Limits, Method 8015C and Lab SOP.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).
Internal standards verification	Every field sample, standard, and QC sample.	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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision/ Representative-ness	Laboratory in-house method manual to be followed for acceptance criteria.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogate spike	All field and QC samples.	Per DoD QSM Appendix C Limits, Method 8015C and Lab SOP.	For QC and field samples, correct problem, then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision/ Representative-ness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per DoD QSM Appendix C Limits, Method 8015C and Lab SOP. MSD or Matrix Duplicate: RPD of all analytes ≤30%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/ Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.4 (DoD and DOE 2021).

DQI data quality indicator
MB method blank
PQO project quality objective
RPD relative percent difference

Table B-7: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-FID and GC-MS	Change gas purifier.	N/A.	Visually inspect if traps are changing color.	Every 6–12 months	No moisture	Replace indicating traps.	Analyst or certified instrument technician	EFGS-T-GCS-SOP40900, EFGS-T-VOA-SOP41119
	Change syringes/syringe needles.	N/A.	Visually inspect for wear or damage.	Every 3 months	N/A	Replace syringe if dirt is noticeable in the syringe.	Analyst or certified instrument technician	
	Change inlet liner, liner O-rings, and inlet septum.	N/A.	Visually inspect for dirt or deterioration.	Weekly for liner Monthly for O-rings Daily for septum	N/A	Replace and check often.	Analyst or certified instrument technician	
	Change front-end column.	N/A.	Check peak tailing, decreased sensitivity, retention time changes, etc.	Weekly, monthly, or when needed	N/A	Remove 1/2 to 1 meter from the front of the column when experiencing problems.	Analyst or certified instrument technician	
	Clean injector ports.	N/A.	N/A.	As needed	N/A	N/A.	Analyst	
	Replace trap on purge-and-trap systems.	N/A.	N/A.	Bi-monthly or as needed	N/A	N/A.	Analyst	
	Replace columns.	N/A.	N/A.	If chromatograms indicate possible contamination	N/A	N/A.	Analyst	

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-FID	Replace detector jets.	N/A.	N/A.	As needed	N/A	N/A.	Analyst	EFGS-T-GCS-SOP40900
	Replace hydrocarbon traps and oxygen traps on helium and hydrogen gas lines.	N/A.	N/A.	Every 4–6 months	N/A	N/A.	Analyst	
	Replace chemical trap.	N/A.	N/A.	Yearly or as needed	N/A	N/A.	Analyst	
	Replace converter tube in gas purifier system.	N/A.	N/A.	Yearly or as needed	N/A	N/A.	Analyst	
GC-MS	Change tune MSD, check the calibration vial, and replace the foreline pump oil.	N/A.	Visually inspect and monitor the fluid becoming discolored.	As needed or every 6 months	In accordance with manufacturer's recommendation or lab SOP	Keep plenty of PFTBA; refill the vial and check the fluid; change when the fluid becomes discolored.	Analyst or certified instrument technician	EFGS-T-VOA-SOP41119
	Run tuning program to determine if source is functioning properly.	N/A.	N/A.	Daily	N/A	Cool system, vent, disassemble, and clean.	Analyst	
	N/A.	Tune instrument.	N/A.	Daily or every 12 hours	Per method	Liner and septa are replaced; tune file used is manually adjusted.	Analyst	
	Vacuum rough pump oil level is checked.	N/A.	N/A.	Every 4–6 weeks	N/A	Add oil if needed.	Analyst	
	Replace/refill carrier gas line oxygen and moisture traps.	N/A.	N/A.	Yearly or as needed	N/A	N/A	Analyst	

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
Water Bath (Precision Microprocessor controlled)	Check instrument connections, water level, and thermometer.	Measure water temperature against a calibrated thermometer.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified instrument technician	EFGS-Q-QD-SOP2831
Drying Oven	Thermometer indicator.	Measure oven temperature against a calibrated thermometer.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified instrument technician	EFGS-Q-QD-SOP2831
Analytical Balance	Check digital LCD display and ensure a flat base for the Instrument.	Calibrate against verified (NIST) mass.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified instrument technician	EFGS-Q-QD-SOP2710
pH Meter	Check LCD display and pH probe.	3-point calibration using known standards.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	±0.05 units	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified manufacture instrument technician	EFGS-T-WC-SOP41204

Note: No instrument and equipment maintenance, testing, and inspection criteria for geotechnical and petrographic analyses.
LCD liquid crystal display
N/A not applicable
NIST National Institute of Standards and Technology
PFTBA perfluorotributylamine
[a] See Analytical SOP References table (Table F-5).

Table B-8: Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-MS EPA Method 8260/ CALUFT	Tuning	Prior to ICAL and at the beginning of each 12-hour period	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-VOA-SOP41119
	Breakdown check (DDT-Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses and should not exceed a tailing factor of 2.	Correct problem, then repeat breakdown checks.	Lab Manager/ Analyst or certified instrument technician	
	Minimum 5-point ICAL for linear calibration Minimum 6-point ICAL for quadratic calibration	Prior to sample analysis	RSD for each analyte ≤15% or least square regression ≥0.995. Non-linear least squares regression (quadratic) for each analyte ≤0.995.	Correct problem then repeat ICAL.	Lab Manager/ Analyst or certified instrument technician	
	Second source calibration verification	After ICAL	All analytes within ±20% of expected value.	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat ICAL.	Lab Manager/ Analyst or certified instrument technician	
	RT window position for each analyte and surrogate	Once per ICAL	Position will be set using the midpoint standard for the ICAL.	N/A	Lab Manager/ Analyst or certified instrument technician	
	RRT	With each sample	RRT of each target analyte in each calibration standard within ±0.06 RRT units of ICAL.	Correct problem, then reanalyze all samples analyzed since the last RT check. If it fails, then rerun ICAL and samples.	Lab Manager/ Analyst or certified instrument technician	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-MS EPA Method 8260/ CALUFT (cont'd)	CCV	Daily, before sample analysis, unless ICAL performed on the same day and after every 10 samples and at the end of the analysis sequence	All analytes within ±20% of expected value (%D). All reported analytes and surrogates within ±50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-VOA-SOP41119
	IS	Each CCV and sample	RT ±10 seconds from RT of the ICAL mid-point standard. EICP area within -50% to +100% of area from IS in ICAL mid-point standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed during failure is mandatory.	Lab Manager/ Analyst or certified instrument technician	
GC-FID EPA Method 8015C	Minimum 5-point ICAL for linear calibration Minimum 6-point ICAL for quadratic calibration	Prior to sample analysis	RSD for each analyte ≤20% or least square regression ≥0.995. Non-linear least squares regression (quadratic) for each analyte ≤0.995.	Correct problem then repeat ICAL.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-GCS-SOP40900
	Second source calibration verification	Once after each ICAL	Analytes within ±20% of expected value (initial source), and within established RT windows.	Correct problem and verify second source standard. Rerun second source verification. If fails, correct problem and repeat ICAL.	Lab Manager/ Analyst or certified instrument technician	
	RT window width	At method set-up and after major maintenance	RT width is ±3 times standard deviation for each analyte RT from 72-hour study. For TPH-d: calculate RT based on C12 and C25 alkanes.	N/A.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-GCS-SOP40900
	Establishment and verification of the RT window for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for establishment of RT; and with each CCV for verification of RT	Using the midpoint standard or the CCV at the beginning of the analytical shift for RT establishment; and analyte must fall within established window during RT verification.	N/A.	Lab Manager/ Analyst or certified instrument technician	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-FID EPA Method 8015C (cont'd)	Run second source calibration verification (ICV)	ICV: Daily, before sample analysis, unless ICAL performed on the same day	All analytes within ±20% of expected value (%D).	Correct problem and rerun ICV. If fails, repeat ICAL.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-GCS-SOP40900
	CCV	Daily, before sample analysis, unless ICAL performed on the same day and after every 10 samples and at the end of the analysis sequence	All analytes within ±20% of expected value (%D).	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/Analyst or certified instrument technician	
Water Bath	Measure water temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/ Analyst or certified instrument technician	EFGS-Q-QD-SOP2831
Drying Oven	Measure oven temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/ Analyst or certified instrument technician	EFGS-Q-QD-SOP2831
Analytical Balance	Calibrate against verified (NIST) mass	Daily or prior to analyzing samples	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/ Analyst or certified instrument technician	EFGS-Q-QD-SOP2710
pH Meter	Run a minimum 3-point calibration; run CCV	Daily or prior to analyzing samples; one CCV for every 10 samples	±0.05 unit.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/ Analyst or certified instrument technician	EFGS-T-WC-SOP41204

Note: No instrument calibration procedures for geotechnical and petrographic analyses.

%D	percent difference	N/A	not applicable
CCV	continued calibration verification	NIST	National Institute of Standards and Technology
DDT	dichlorodiphenyltrichloroethane	RRT	relative retention time
ICV	initial calibration verification	RSD	relative standard deviation
IS	internal standard	RT	retention time

Table B-9: Data Verification and Validation (Steps I and IIa/IIb) Process

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/IIa/IIb [a]	Internal/ External
Laboratory system audits	Determine whether the laboratory holds a current DoD Environmental Laboratory Accreditation Program certification for all analyses to be performed for the project.	Project Chemist (Navy consultant)	Step I	Internal
Field procedures	Determine whether field procedures are performed in accordance with this SAP and prescribed procedures.	QA Program Manager (Navy consultant)	Step I	Internal
Field logbook and notes	Review the field logbook and any field notes on a weekly basis and place them in the project file. Copies of the field logbook and field notes will be provided to the Navy consultant contract task order manager and included in the Field Audit Report.	Field Manager (Navy consultant)	Step I	Internal
CoC forms	Review CoC completed forms and verify them against the corresponding packed sample coolers. A copy of each CoC will be placed in the project file. The original CoC will be taped inside the cooler for shipment to the analytical laboratory.	Project Chemist (Navy consultant)	Step I	Internal
Sampling analytical data package	Verify all analytical data packages for completeness prior to submittal of the data to the Navy consultant.	Laboratory Project Manager (Eurofins)	Step I	External
Analytes	Determine whether all analytes requested were analyzed and reported on by the laboratory.	Project Chemist (Navy consultant)	Step IIa	Internal
CoC and field QC logbook	Examine data traceability from sample collection to project data generation.	Project Chemist (Navy consultant)	Step IIa	Internal
Sampling plan	Determine whether the number and type of samples specified in the SAP were collected and analyzed.	Project Chemist (Navy consultant) & Field Manager (Navy consultant)	Step IIb	Internal
Field QC samples	Establish that the number of QC samples specified in the SAP were collected and analyzed.	Project Chemist (Navy consultant)	Step IIb	Internal
Project quantitation limits and data qualifiers	Establish that sample results met the project quantitation limits.	Project Chemist (Navy consultant)	Step IIb	Internal

[a] IIa Compliance with methods, procedures, and contracts. See Table 10, page 117, UFP-QAPP manual, V.1 (DoD 2005).
IIb Comparison with measurement performance criteria in the SAP. See Table 11, page 118, UFP-QAPP manual, V.1 (DoD 2005).

Appendix C – Attachment 1. Sample Log Form

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Attachment 1 Sampling Log Form

Sample Log

Site Name:
Holding Tank/Leach Tank TPH Quantification Sampling

MIS Sample ID: _____

Replicate Sample: Yes No

Start Date/Time _____ End Date/Time _____

Weather Conditions: Clear Overcast Rain Wind Other (describe): _____

Media: Soil

Approx. DU Volume (cubic ft): _____ (Combined volume of trucks sampled)

Total Number of MI Increments: _____ (Should equal sum of Increments below)

Manifests for trucks sampled as part of MIS:	Number of MIS Increments per truck
1)	
2)	
3)	
4)	
5)	
6)	
7)	
8)	
9)	
10)	
11)	
12)	
13)	

Sampling Equipment: Dedicated Sampling Scoop (Y) Dedicated T-Sampler (Y)

Soil Description: _____

Field Processing: _____

Approximate MIS Sample Volume/Mass: _____

Sample Container: _____

Number of Replicates Collected: _____ (Collect 3 replicates, when applicable)

Field Observations (debris, staining, odors, PID readings, etc.):
MIS Headspace Concentration =

Photographs: Yes No

Comments

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