Final NPS Preliminary Assessment and Site Inspection Report

Sitka National Historical Park

Indian River Asphalt Plant
EDL Number 5AKR2141

Prepared by
Michael Baker International/Ahtna Engineering Services, LLC

3/26/2019

Signatories:

Mary Miller Superintendent	Tracy A. Lagua, Acting for Brinnen Carter Park Chief of Natural Resources	William F. Heubner NPS Project Engineer
Signature	Signature	Signature
3-24-19 Date	3-26-2019 Date	3/26/19 Date
By signing above, the signatories verify that they understand and concur with the information and recommendations presented herein.		

Table of Contents

Figures

Tables

Appendicies

List of Abbreviations and Acronyms

AAC	Alaska Administrative Code
ADEC	Alaska Department of Environmental Conservation
Ahtna	Ahtna Engineering Services, LLC
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cfs	cubic feet per second
DRO	diesel range organics
DQO	Data Quality Objective
MBI	Michael Baker International
mg/kg	milligrams per kilogram
MTG	migration to groundwater
NCP	National Oil and Hazardous Substances Pollution Contingency Plan (AKA, National Contingency Plan)
NPS	National Park Service
PA	Preliminary Assessment
PA/SI	Preliminary Assessment/Site Inspection
PAH	polycyclic aromatic hydrocarbon
RRO	residual range organics
SAP	Sampling and Analysis Plan
ppm	parts per millions
SI	Site Inspection
Site	Indian River Asphalt Plant Site
SITK	Sitka National Historical Park
SPLP	Synthetic Precipitation Leaching Procedure
SVOC	semivolatile organic compound
S&W	Shannon and Wilson, Inc.
TAH	total aromatic hydrocarbons
TAqH	total aqueous hydrocarbons
TCLP	toxicity characteristic leaching procedure
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

(This Page Intentionally Left Blank)

1. Introduction

This document serves as the Preliminary Assessment/Site Inspection (PA/SI) report for the Indian River Asphalt Plant Site (Site) in the Sitka National Historical Park (SITK), Alaska. Ahtna Engineering Services, LLC (Ahtna) is conducting this work under subcontract to Michael Baker International (MBI) under National Park Service (NPS) notice-to-proceed number 140P9718Q0026. The Alaska Department of Environmental Conservation (ADEC) File Number is 1525.38.055, and the Site Environmental and Disposal Liabilities number is 5AKR2141. Fieldwork was conducted at the site on August 23, 2018. Sampling was conducted by Ahtna with oversight by Bill Heubner, Project Engineer for the NPS.

The site is a former asphalt hot plant located on the northeast bank of the Indian River, where surplus asphalt and debris from the plant were buried. Significant storm events and river flooding in recent years have given way to erosion in the area, exposing the buried materials. Reports of sheen on the river, which is likely associated with this site, have prompted investigation of the potential environmental concerns (NPS, 2018).

The purpose of this PA/SI is to define:

- The potential for petroleum impacts from the remaining asphalt in the ground at the Site; and
- Assess whether compounds from the asphalt are or are not currently impacting the waters of the Indian River.

The Site is located within the boundaries of SITK (Figures 1 and 2), in Sitka, Alaska. Section 3 lists detailed location information.

The PA/SI was combined to minimize costs associated with travel to the site.

This report includes a summary of field activities and sampling results, a discussion of site history and previous investigations, an assessment of exposure pathways, and conclusions and recommendations.

1.1. CERCLA and NPS Authority

NPS is authorized under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 United States Code Section 9601 et seq., to respond as the lead agency to a release or a threatened release of hazardous substances. The NPS is also the lead response agency for a release or threatened release of any pollutant or contaminant that may present an imminent and substantial danger to public health or the environment on NPS land.

CERCLA's implementing regulations, codified in the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 CFR Part 300, establish the framework for responding to such releases and threatened releases. The NCP prescribes two similar processes for responding to releases; removal actions and remedial actions (See NCP Sections 300.400 through 300.440). Under either process, the initial step is to perform a Preliminary Assessment (PA). If the PA does not 1) conclude that a release or threat of release exists, 2) confirm whether the contaminants releases include "hazardous substances," or 3) determine whether these contaminants pose a threat to public health or the environment, then environmental sampling is warranted under a Site Inspection (SI). See NCP Sections 300.410 and 300.420.

At sites that clearly warrant a SI or at sites that are remote and/or have high mobilization costs, the PA and SI may be combined into one continuous field event in order to reduce costs and prevent repetitive tasks (United States Environmental Protection Agency [USEPA], 1999).

The purpose of this PA/SI is to investigate suspected petroleum contamination potentially associated with asphalt production activities at the Site in order to determine whether a release or potential release of hazardous substances, pollutants, or contaminants has occurred or could occur. This PA/SI also serves to provide the basis for the NPS to determine whether conditions at the Site warrant further investigation or a no further action determination (i.e., poses no risk to human health or the environment). See NCP Sections 300.410 and 300.420. Evaluations are focused on past and present practices and processes related to the storage, use, and disposal of hazardous substances at the Site. Emphasis is placed on activities that routinely or non-routinely may have led or may lead to releases of hazardous substances into the environment.

2. Sampling and Analysis Plan and Data Quality Objectives Summary

This section provides a summary of the Sampling and Analysis Plan (SAP) and Data Quality Objectives (DQOs) that were used to guide the collection of data under this PA/SI.

2.1. Sampling and Analysis Plan

Fieldwork for this PA/SI was conducted in accordance with the ADEC-approved SAP for the Site, dated August 10, 2018. The SAP is attached as Appendix E.

2.2. Data Quality Objectives

The DQOs developed for the SAP were chosen to allow for comparison of petroleum constituents in pore water and from asphalt leachate to ADEC groundwater and surface water cleanup levels, promulgated in Title 18 of the Alaska Administrative Code (AAC), Chapter 75 (18 AAC 75) and 18 AAC 70, respectively. A detailed discussion of the DQOs is provided in Section 4.0 of the SAP, appended to this report (Appendix E). A summary of the data quality review is presented in Section 3.3 of this report.

2.3. Field Activities and Deviations from the Sampling and Analysis Plan

This section provides a summary of the field activities that were conducted and deviations from the SAP that were necessitated due to field conditions encountered. Sampling activities were conducted on August 23, 2018. Work was conducted on behalf of the NPS by Alexa FitzGerald, MA, an Ahtna environmental scientist. She meets the definition of a "qualified environmental professional," as defined in 18 AAC 75.333 (ADEC, 2018). Bill Heubner, project engineer for the NPS, oversaw the fieldwork. Selected photographs are provided in Appendix A and field notes in Appendix B.

2.3.1. Pore Water Sampling

Three locations on the beach were identified for pore water sampling (Figure 2). The 1/8-inch diameter, 14-inch long MHE® pore water sampler was inserted 10 to 12 inches into the soil/sediment in no more than three inches of water (depending on wave action) at the water/shoreline interface. Samples were collected for volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), per the SAP. Sample location PW-1 was located at the site, PW-2 in an upgradient area, and PW-3 was down-gradient of the site. A duplicate sample (SITK-18-PW-4, collocated with SITK-18-PW-X) was collected for each analysis. These samples were labeled and shipped to the analytical laboratory in Tacoma, Washington, to be analyzed for VOCs by USEPA Method 8260C and SVOCs by USEPA Method 8270D. Laboratory results are discussed in Sections 4 and 5.

2.3.2. Asphalt Sampling

Two pieces of asphalt were collected from the site (SITK-18-AS-1 and -3). Per instructions from the project laboratory, the asphalt was broken into small pieces and placed in sample jars in a manner to minimize headspace (Figures 2 and 3). One duplicate sample was collected (SITK-18-AS-2, duplicate of SITK-18-AS-1). The samples were labeled and shipped to the analytical laboratory in Tacoma, Washington, for Synthetic Precipitation Leaching Procedure (SPLP) extraction by USEPA Method 1312

and the extract analyzed for VOCs by USEPA Method 8260C and SVOCs by USEPA Method 8270D. Laboratory results are discussed in Sections 4 and 5.

2.3.3. Deviations from the Sampling and Analysis Plan

The jars provided for SVOC analysis for were improperly preserved. This deviation is discussed further in the Data Quality Review in Appendix D.

2.4. Data Quality Review Summary

Based on the data review completed, no data were rejected. Data qualifiers were assigned due to poor field duplicate precision. All analytical data is considered usable for the purpose of evaluating the presence or absence and magnitude of the suspected site contaminants. The laboratory report is provided in Appendix C, and a detailed data quality review is provided in Appendix D, along with ADEC Laboratory Data Review Checklists.

3. Site Description, Operational History, and Waste Characteristics

This section summarizes all the known environmental information and historical activities that have occurred at the Site, as well as information regarding waste characteristics at the Site. Ahtna used the following information sources to evaluate the Site:

- Previous Phase II site investigation reports;
- Site information provided by Bill Heubner, NPS Regional Engineer for Contaminated Sites;
- Site information provided by Brinnen Carter, Chief of Resources, SITK; and
- Data gathered during the subject SI.

3.1. Site Description

The Site is located on the northeast bank of the Indian River that encompasses an area of approximately 3/4 acre within the boundary of SITK (Figure 2). The mailing address for the park is 103 Monastery St., Sitka, AK 99835. The Site is located at latitude 57 degrees, 2 minutes, 50 seconds North and longitude 135 degrees,18 minutes, and 39 seconds West; and is located in the northwest quarter of Section 6, Township 56 South, Range 64 East, of the Copper River Meridian.

The Site is bordered by SITK lands, Sawmill Creek Road to the north, Crescent Bay to the south, Arrowhead Trailer Court to the east and tidal flats of the Indian River to the west. There are residential, industrial and undeveloped areas surrounding SITK (Shannon & Wilson, Inc. [S&W], 1995). An established trail system runs through SITK and the Site, as well as a memorial plaque of the 1804 Battle of Sitka, which occurred between the Tlingit tribe in Sitka and the Russian explorers. The memorial plaque receives visitors regularly. The SITK NPS Visitor Center offers the public guided walking tours to the memorial from May through September.

The nearest residential structures are the homes in the Arrowhead Trailer Court, and there is a wood fence separating the Park from the Arrowhead Trailer Court homes. Access to the Site can be gained via a trail off of Sawmill Creek Road, the footbridge that spans the Indian River, or at the southern end of the fenced-off Arrowhead Trailer Court residential area.

3.2. Operational History

Private interest groups, as well as the United States Army and Navy, began extracting gravel from the Indian River in the 1940s. In the late 1950s, the Morrison-Knudsen Company established and began operating the Indian River Asphalt Plant to support railway construction in the City of Sitka. The plant used gravel from the river as aggregate to produce the pavement. An area 60-feet deep was dredged at the mouth of the Indian River, and approximately 1.5 million cubic yards of material was dredged from the sea. After several years of operation, the plant was closed and debris, including machinery, metal, cable, and old asphalt, was buried at the Site and abandoned (S&W, 1995). Currently, the Site is mostly overgrown with forest; however, erosion during storm events has exposed the buried debris associated with the former asphalt plant (NPS, 2018).

3.3. Previous Investigations and Response Actions

Reports of a sheen in the Indian River at the base of the Site have been documented since 1990. In May of 1993, Ms. Barbara Reilly of the U.S. Army Corps of Engineers collected one soil sample from the Site and submitted it for diesel range organics (DRO), gasoline range organics (GRO), total recoverable petroleum hydrocarbons (TPH) and toxicity characteristic leaching procedure (TCLP) for VOCs and TCLP for SVOCs. Results indicated DRO at a level of 1,820 parts per million (ppm) and TPH at 3,400 ppm, which exceed ADEC soil cleanup levels at that time (DRO exceeds current ADEC soil cleanup levels per 18 AAC 75, TPH is not regulated). No other results exceeded cleanup levels (S&W, 1995).

S&W completed a Level 2 Environmental Site Assessment in October 1994 at the former Indian River asphalt plant, "*to identify and characterize, to the extent possible, the source of the oil saturated sediment layer and vegetative mat that have been exposed from an eroding bank along the Indian River, and to outline remedial actions for the site*" (S&W, 1995). During this investigation, ten test pits were dug at the Site and 33 soil samples were collected for DRO, residual range organics (RRO) and VOC analysis. Samples collected from the exposed bank were also analyzed for arsenic, cadmium, chromium, lead, and mercury.

DRO was detected in 24 of the 33 soil samples, with concentrations ranging from 12 milligrams per kilogram (mg/kg) to 6,100 mg/kg; 6 of the 24 samples exceeded the18 AAC 75 Method Two, Over 40 Inches, DRO migration to groundwater (MTG) soil cleanup level of 230 mg/kg. RRO was detected in 27 of the 33 soil samples, with concentrations ranging from 25 mg/kg to 8,700 mg/kg, all below the MTG cleanup level of 11,000 mg/kg. None of the samples exceeded cleanup levels for VOC constituents, though one sample had an ethylbenzene concentration of 0.12 mg/kg. Arsenic, chromium and lead were reported in all of the 11 analyzed test pit samples. Concentrations ranged from 3.5 mg/kg to 12 mg/kg for arsenic, 15 mg/kg to 39 mg/kg for chromium, and 6.3 mg/kg to 3,500 mg/kg for lead (the elevated lead reading of 3,500 mg/kg appeared to be limited in quantity and only in one sample in one test pit). There was one detection of mercury at 0.12 mg/kg. For the two samples collected from the bank, arsenic was detected at 6.3 mg/kg and 26 mg/kg; chromium at 33 mg/kg and 39 mg/kg; and lead at 5.6 mg/kg and 10 mg/kg. (NPS, 2018). The metals results were found to be consistent with background levels.

Remediating the high level of exceedances throughout the Site would require the removal of approximately 400 cubic yards of contaminated soil. However, due to the high cost and potential damage to this area of SITK, the NPS and ADEC concluded that it would be in the best interest of both parties to conduct monitoring of the Site rather than a removal effort. Water quality monitoring efforts conducted at the site in the early 2000s detected no significant contamination, and in 2006, the NPS and ADEC agreed to stop monitoring at the site.

Much of the site (approximately 50 feet of beach area) has eroded into the waters of the Indian River and estuary since 1995, and buried debris at the site has become exposed (NPS, 2018). In August 2017, the NPS Alaska Region's Environmental Management Program Coordinator conducted a site visit and collected two samples of exposed asphalt tar that was oozing from a large mass on the beach. The samples were sent to a certified laboratory for SVOC analysis. The results indicated exceedances of ADEC Method Two MTG cleanup levels for several of the SVOC constituents. Table 3-1 summarizes

SVOC compounds exceeding current ADEC soil cleanup levels. The large mass was removed by NPS personnel and disposed of offsite in September 2017. Small pieces of asphalt remain at the site and are exposed periodically at times of high tide and high river events (NPS, 2018).

TABLE 3-1: ASPHALT SVOC EXCEEDANCES

Analyte	Maximum Detected Concentration in mg/kg	ADEC Cleanup Level in mg/kg[1]
Benzo[a]anthracene	1.3	0.28
2,4-Dinitrotoluene	7.7	0.024
Dibenzofuran	5.7	0.97
1-Methylnaphthalene	37	0.41
2-Methylnaphthalene	48	1.3
Naphthalene	21	0.038
Phenanthrene	47	39

Notes:

ADEC – Alaska Department of Environmental Conservation

mg/kg – milligrams per kilogram

SVOC – semivolatile organic compound

3.4. Waste Characteristics

S&W, the consultant who conducted the Environmental Site Assessment in 1995 on behalf of NPS, concluded that approximately 400 cubic yards of DRO- and RRO-contaminated soil was present at the Site. Contaminants of concern at the Site for purposes of the 2018 PA/SI are VOCs and SVOCs.

3.5. Known and Potential Source Areas

The source for the petroleum impacts at this site is operations at the former asphalt plant. The location of specific features (e.g. aboveground storage tanks) are not specifically known; only remaining pieces of asphalt are found at the site. The 1995 S&W report showed four locations with DRO above MTG cleanup levels for DRO (Figure 4). Test pit TP-1 (1,900 mg/kg) is within the zone that has started sloughing off into the Indian River/Sitka Sound. TP-9 (840 mg/kg) is at the edge of the current bluff at the site, and TP-6 (440 mg/kg DRO) and TP-10 (6,100 mg/kg DRO) are still present.

4. Exposure Pathway and Environmental Hazard Assessment

This section provides an evaluation of the potentially contaminated media, associated exposure pathways, and sensitive environments that are known and/or suspected at the Site. An evaluation of the potential for a hazardous substance release to each media is also presented.

4.1. Groundwater

Groundwater at the site is assumed to be shallow, to generally flow towards the Indian River and Sitka Sound, and to have fluctuating levels with tidal variation near the shore. Groundwater quality discharging from the site was assessed by collecting pore water samples at the land/water interface.

4.1.1. Local Geologic and Hydrogeologic Setting

At the head of Indian River and its tributaries are the steep mountain side slopes and cirque walls, formed during local alpine glaciation that shed the source rocks for surficial deposits. The deposits consist of primarily graywacke, schist, and phyllite, which cover the majority of Sitka, and include alluvium on Indian River's floodplain, estuary, and stream terrace. Ablation till exists on the lateral moraine, and beach sands and gravel are the primary geology on the uplifted beach and uplifted beach meadow where the project site is located (NPS, 2015).

The Indian River forms an estuary as it enters into the ocean just below the Site. North of SIKA are the Baranof Island Mountains, where the Indian River originates. From there it flows into Sitka Sound, between Crescent and Jamestown Bays. A large U-shaped post-glacial valley watershed exists there, encompassing approximately 12.3 square miles with an elevation range of 0 to 3,800 feet above mean sea level. The river and upper basin alpine regions drain into the watershed toward the ocean. The valley floor consists of a wide, flat area, covered by muskeg and a forest of primarily Sitka spruce and western hemlock (NPS, 2016). The project site is densely populated with alder, Sitka spruce, and undergrowth (S&W, 1995).

Local drinking water for the City of Sitka is supplied by surface water from Blue Lake. The Indian River was once considered a drinking water supply for the city; however, it is no longer a drinking water source (City and Borough of Sitka, 2015). No known drinking water wells exist in the vicinity of the Site (Bill Heubner, 2018). It should be noted that the proximity of the Site to salt water would render it unsuitable for the placement of drinking water wells.

4.1.2. Hazardous Substance Release Determination

It is suspected that groundwater at the Site is contaminated by DRO; however, based on pore water sampling results (Table 1), groundwater does not appear to be impacting Site surface water. Groundwater will never be used as a drinking water source. However, sheen has occasionally been observed in surface water, suggesting further assessment of groundwater is needed.

4.2. Surface Water

Pore water samples were collected at the land/water interface to assess potential impacts to surface water. No VOC or SVOC contaminants were detected at concentrations exceeding ADEC cleanup levels. Total aromatic hydrocarbons (TAH) and total aqueous hydrocarbons (TAqH) concentrations were below ADEC

surface water criteria set forth in 18 AAC 70 (Table 1). If sheen continues to be observed at the base of the bluff, further assessment of surface water may be required.

4.2.1. Local Hydrologic Setting

The Indian River watershed is characterized by steep topography and consists of shallow soils with high drainage density. The watershed has a rapid response to rainstorms, which can cause large daily fluctuations in stream flow. Hydrologic calculations indicate that the height of runoff occurs within 6 hours of a storm event, and nearly all rainfall runs through the watershed within 12 to 24 hours. River flow ranges from approximately 20 cubic feet per second (cfs) to 6400 cfs. River discharge is generally at its peak in September and October and tends to decline throughout winter and early spring. Snowmelt at high elevations results in moderate flow increases in May and June, while minimum flows are most common in December, March, and July (NPS, 2016).

4.2.2. Drinking Water Intakes

As stated in Section 4.1.1, local drinking water for the City of Sika is supplied by surface water from Blue Lake. The Indian River was once considered a drinking water supply for the city, however it is no longer a drinking water source (City and Borough of Sitka, 2015).

4.2.3. Local Fisheries

Indian River is an anadromous stream. Many fisheries exist in the area of Sitka and southeast Alaska.

4.2.4. Sensitive Environments

The Site is within a national park unit therefore is considered a sensitive environment.

4.2.5. Hazardous Substance Release Determination

Groundwater discharging to surface water has been tested and no VOC or SVOC analytes were detected. Calculated TAH and TAqH concentrations were assessed in surface water and are below applicable criteria (Table 1).

4.3. Soil

Soil was not directly assessed during this effort, but DRO impacts above MTG soil cleanup levels were observed in 1995. Asphalt in the soil was assessed for its potential to further impact soil quality. Laboratory analysis of the asphalt showed various chemicals to be present in the asphalt itself (Table 3-1). However, SPLP extraction and analysis of VOC and SVOC contaminants suggest that further impacts to soil and groundwater through precipitation will not occur at the site. Further removal of the asphalt remaining at the site for the purposes of mitigating further impacts to the environment should not be required. Note that this does not address any aesthetic or other considerations that may be evaluated by the NPS.

In 1995, S&W found soil with DRO concentrations exceeding ADEC MTG cleanup levels at the Site. Further assessment of the soil is needed to assess whether current DRO levels are still above regulatory clean up levels and to evaluate the lateral extent of the DRO impacts.

4.3.1. Potential Receptors

The primary receptors are as follows:

- SITK visitors/recreational users – on average, 100 people visit the park on a daily basis.
- NPS personnel accessing the site – one to two NPS personnel (rangers, maintenance) access the site.
- Nearby residents – a trailer park is present at the eastern border of the park. A fence separates the site and the trailer park.
- Vegetation – trees and other lower lying vegetation are present at the site.
- Invertebrates are likely present in the soil
- Small birds and mammals are likely present that may ingest invertebrates or plants.
- Fish that may encounter contaminated surface water – juvenile and adult salmon (pink, coho, and chum salmon; steelhead; and Dolly Varden char) are present.
- Larger birds (ducks, bald eagles, and ravens) and mammals (land otters) are present that may ingest fish, other birds, small mammals, or plants.
- No cultural resources are present in the immediate area of impacted soil.
- No schools or daycare facilities are located within 200 feet of area(s) of known or suspected contamination.

4.3.2. Sensitive Environments

The Site is within a national park unit and therefore is considered a sensitive environment.

4.3.3. Hazardous Substance Release Determination

Asphalt pieces remaining in the soil were sampled and tested to determine if the remaining asphalt itself poses a risk to further soil and groundwater impact at the site. Results of SPLP extraction, followed by VOC and SVOC analysis, showed the remaining asphalt poses itself little or no risk to further contaminating the soil or groundwater at the site (Table 2).

Soil containing DRO at concentrations above ADEC MTG cleanup levels was present at the site in the area of TP-1, TP-6, TP-9, and TP-10 in 1994 (S&W, 1995). No immediate indications of the contamination were apparent during the 2018 site visit (odors, stressed vegetation).

The levels of DRO and other VOC do not suggest that outdoor air is a complete pathway. No buildings are within 30 feet of the impacted areas.

(This Page Intentionally Left Blank)

5. Conclusions and Recommendations

SPLP analysis of the asphalt remaining at the Site suggests that further impacts to soil and groundwater through precipitation water leaching should not occur.

Pore water sampling demonstrated that TAH, TAqH, and VOC and SVOC contaminants are not significantly impacting surface water.

Soil sampling in 1995 encountered soils with DRO concentrations above the ADEC cleanup level for MTG; however, the horizontal extent of soil impacts and current petroleum levels are unknown. Ahtna recommends installing soil borings near former test pits TP-6, TP-9, TP-10, and to the north and to the east of TP-10. Soil samples should be collected from each boring and analyzed for VOCs, DRO, and polycyclic aromatic hydrocarbons (PAHs).

A monitoring well should be installed near former test pit TP-10 to determine if impacts to groundwater quality exist. Groundwater samples should be analyzed for VOC, DRO, and PAH.

Based on the S&W 1994 site assessment, a soil boring should be advanced near former test pit TP-7 and samples collected for the presence of lead.

While chemical analysis shows that the asphalt at the site will not leach contaminants, to further assess the potential source of the sheen occasionally observed in surface water, we recommend that placing pieces of asphalt from the site in a container then submerging the asphalt in water and observing whether any petroleum sheen in produced.

(This Page Intentionally Left Blank)

6. References

Alaska Administrative Code (AAC) Title 18 Chapter 75 (18 AAC 75). Revised as of October 27, 2018.

City and Borough of Sitka Public Works Department Environmental Division (City and Borough of Sitka). 2015. Drinking Water Quality Report for the Year 2015, Sika, Alaska. (Public Water System AK2 130075). http://www.cityofsitka.com/government/departments/publicworks/documents/DrinkingWaterQualityReport2015.pdf. Accessed May 30, 2018.

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 United States Code Section 9601.

Heubner, Bill. 2018. Personal Communication.

National Oil and Hazardous Substances Pollution Contingency Plan, 40 Code of Federal Regulations, Part 300.

NPS. 2018. Statement of Work: Indian River Asphalt Plant Preliminary Assessment/Site Inspection, Sitka National Historic Park. January 12.

NPS. 2016. Hydraulic Activity, Sitka National Historic Park, Alaska. https://www.nps.gov/sitk/learn/nature/hydrologicactivity.htm. Last updated April 22, 2016. Accessed May 30, 2018.

NPS. 2015. Geologic Formations, Sitka National Historic Park, Alaska. https://www.nps.gov/sitk/learn/nature/geologicformations.htm. Last updated April 14, 2015. Accessed May 30, 2018.

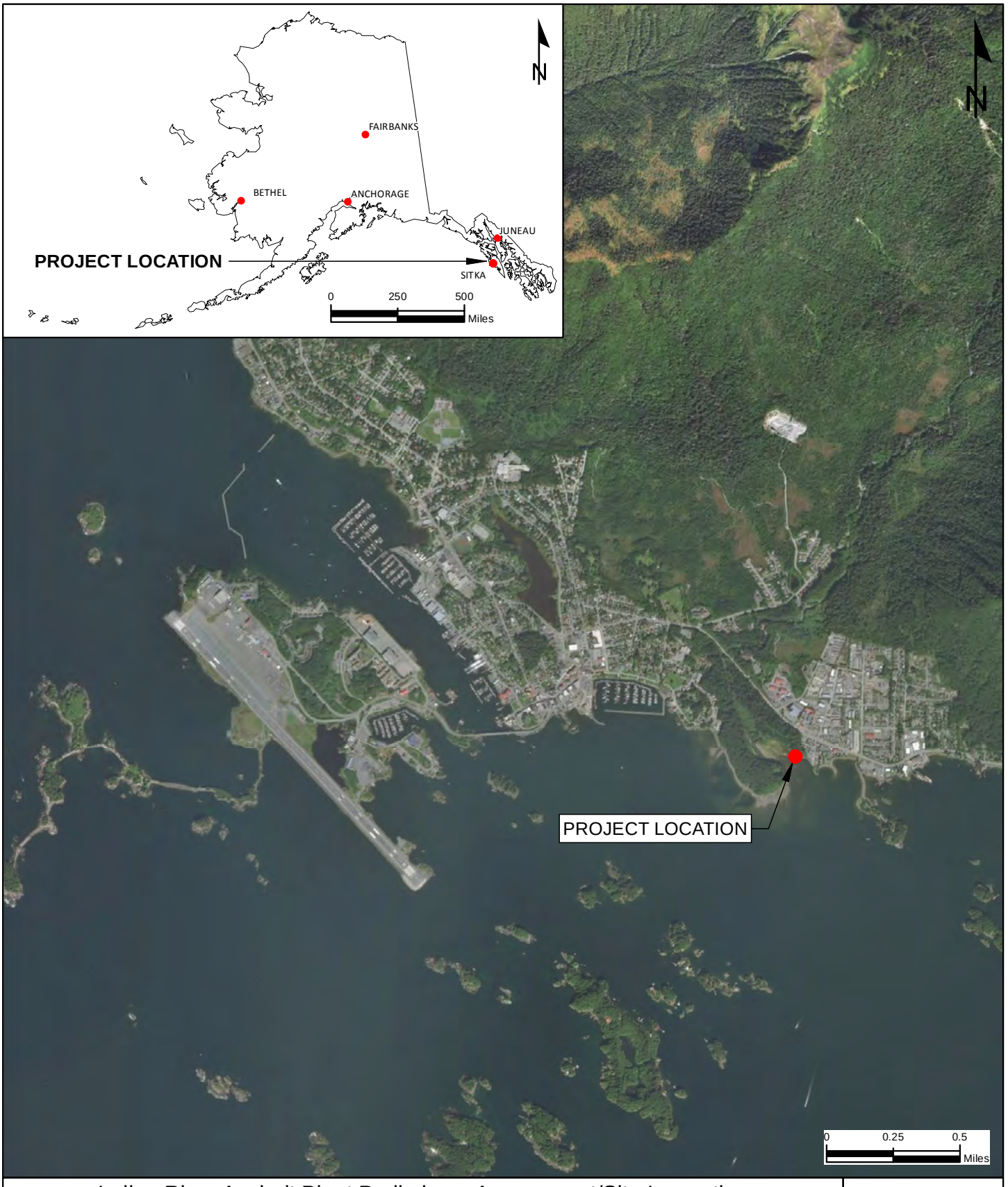
Shannon & Wilson, Inc.. 1995. Environmental Site Assessment, Indian River Asphalt Site, Sitka National Historic Park, Sitka, Alaska. April 7.

USEPA. 1999. Improving Site Assessment: Combined PA/SI Assessments. EPA/540/F-98/038. October.

(This Page Intentionally Left Blank)

Figures

(This Page Intentionally Left Blank)



Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

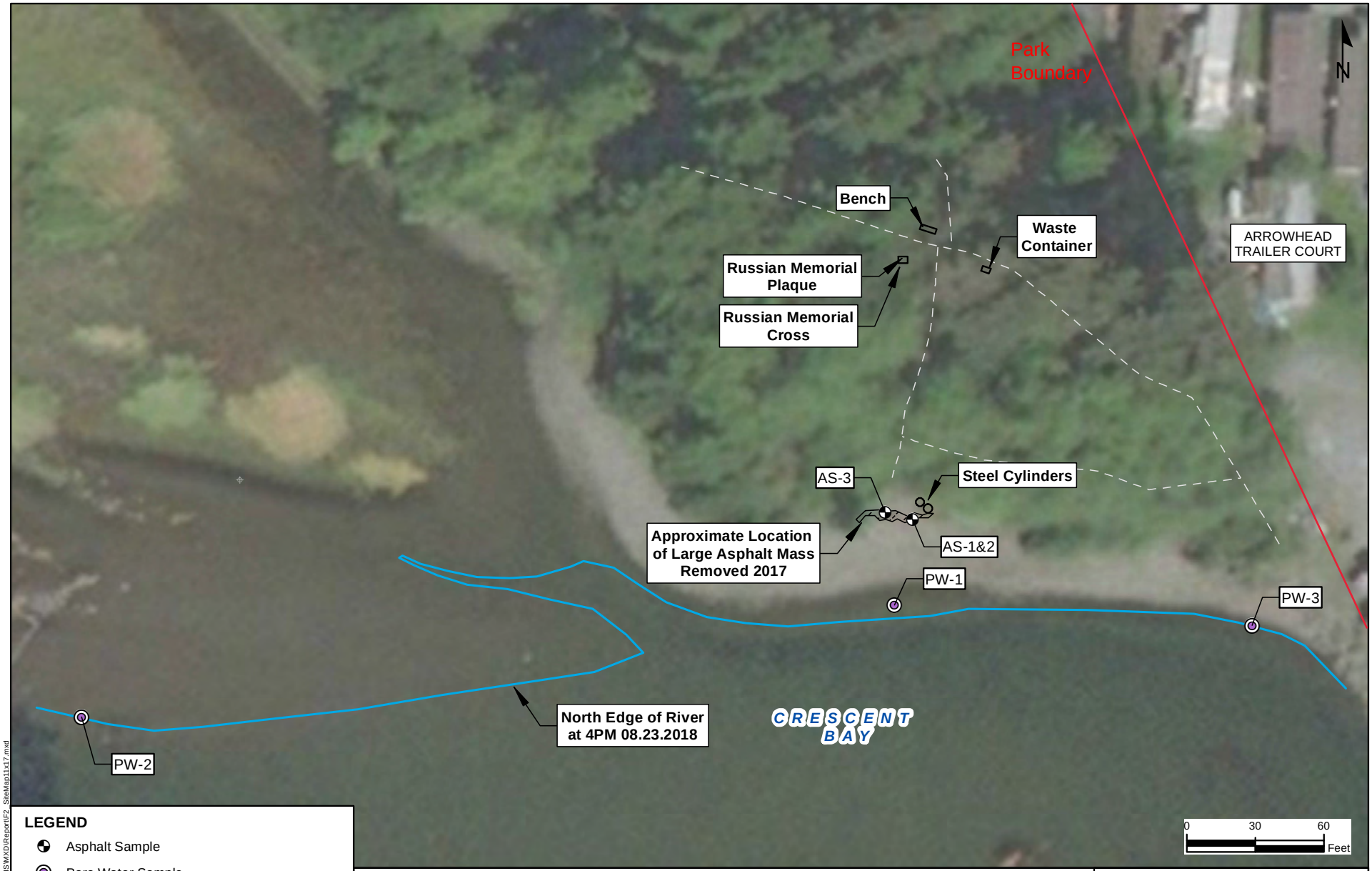
Ahtna Engineering Services, LLC

State & Site Vicinity Map

Project Number:	Figure Number:
20279.011	1
Date: 10/29/2018	
Drafted By: R.F.	

Prepared by rhuelson, 10/29/2018; C:\Users\rfye\Desktop\Sitka\GIS\MXD\Report\F1_State&SiteVicinity.mxd

(This Page Intentionally Left Blank)



LEGEND

- Asphalt Sample
- Pore Water Sample
- Northern Edge of River 8/23/18
- Trail (approx)
- Site Features

Notes:
1. Drawing based on Figure C-1 by Bill Heubner 09.26.2018

Document Path: C:\Users\rfrye\Desktop\Sitka\GIS\MXD\Report\F2_SiteMap11x17.mxd

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

Ahtna Engineering Services, LLC

Site Map

Project Number:	Figure Number:
20279.011	2
Date: 11/6/2018	
Drafted By: R.F.	

(This Page Intentionally Left Blank)

LEGEND

- Asphalt Sample
- Pore Water Sample
- Trail (approx)
- Site Features

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

Ahtna Engineering Services, LLC

Asphalt & Pore Water Sample Locations

Project Number:	Figure Number:
20279.011	3
Date: 10/29/2018	
Drafted By: R.F.	

Prepared by rhuelson, 10/29/2018; C:\Users\rfye\Desktop\Sitka\GIS\MXD\Report\F3_Asphalt&PoreWaterSamples.mxd

(This Page Intentionally Left Blank)

LEGEND

- Test Pit with DRO in mg/kg
- Test Pit with DRO Exceedance in mg/kg
- Asphalt Sample
- Pore Water Sample
- Trail (approx)
- Site Features

Notes:
DRO Diesel Range Organics
mg/kg milligrams per kilogram

1. DRO Exceedance signifies DRO over cleanup level of 230 mg/kg.

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

Ahtna Engineering Services, LLC

Location of 1994 S&W Test Pits

Project Number:	Figure Number:
20279.011	4
Date: 11/6/2018	
Drafted By: R.F.	

Prepared by rhuetson, 11/6/2018; C:\Users\rfrye\Desktop\Sitka\GIS\MXD\Report\F4_1994S&WTestPits.mxd

(This Page Intentionally Left Blank)

Tables

(This Page Intentionally Left Blank)

TABLE 1: PORE WATER ANALYTICAL RESULTS
INDIAN RIVER ASPHALT PLANT
SITKA NATIONAL HISTORICAL PARK, ALASKA

Analytical Method	Sample Name:	Pore Water Samples								ADEC Cleanup Level[1,2]
		SITK-18-PW-1	2X MDL	Field Duplicate SITK-18-PW-4	2x MDL	SITK-18-PW-2	2x MDL	SITK-18-PW-3	2x MDL	
EPA 8260C VOC in µg/L	Benzene	ND (1.0)	1.06	ND (3.0)	1.06	ND (3.0)	1.06	ND (3.0)	1.06	***4.6***
	Toluene	ND (2.0)	0.78	ND (2.0)	0.78	ND (2.0)	0.78	ND (2.0)	0.78	***1,100***
	Ethylbenzene	ND (1.0)	1.00	ND (3.0)	1.00	ND (3.0)	1.00	ND (3.0)	1.00	***15***
	Total-Xylenes	ND (5.0)	1.80	ND (5.0)	1.8	ND (5.0)	1.8	ND (5.0)	1.8	***190***
TAH[3]			4.6		4.6		4.6		4.6	***10***
Other VOC		None Detected								***Various***
EPA 8270D SVOC in µg/L	Acenaphthene	ND (0.41)	0.162	ND (0.40)	0.16	ND (0.39)	0.158	ND (0.39)	0.154	***530***
	Acenaphthylene	ND (1.0)	0.2	ND (1.0)	0.2	ND (0.98)	0.196	ND (0.97)	0.194	***260***
	Anthracene	ND (15)	0.04	ND (15)	0.04	ND (15)	0.04	ND (15)	0.038	***43***
	Benzo(a)anthracene	**ND (1.0)**	0.04	**ND (1.0)**	0.04	**ND (.98)**	0.04	**ND (0.97)**	0.038	***0.30***
	Benzo(a)pyrene	**ND (1.0)**	0.04	**ND (1.0)**	0.04	**ND (.98)**	0.04	**ND (0.97)**	0.038	***0.25***
	Benzo(b)fluoranthene	ND (1.0)	0.102	ND (1.0)	0.1	ND (.98)	0.098	ND (0.97)	0.096	***2.5***
	Benzo(g,h,i)perylene	**ND (1.0)**	0.102	**ND (1.0)**	0.1	**ND (.98)**	0.098	**ND (0.97)**	0.096	***0.26***
	Benzo(k)fluoranthene	**ND (1.0)**	0.04	**ND (1.0)**	0.04	**ND (.98)**	0.04	**ND (0.97)**	0.038	***0.8***
	Chrysene	ND (0.61)	0.34	ND (0.60)	0.34	ND (0.59)	0.34	ND (0.58)	0.32	***2.0***
	Dibenzo(a,h)anthracene	**ND (0.61)**	0.04	**ND (0.60)**	0.04	**ND (0.59)**	0.04	**ND (0.58)**	0.038	***0.25***
	Fluoranthene	ND (3.0)	0.3	ND (3.0)	0.3	ND (3.0)	0.3	ND (2.9)	0.3	***260***
	Fluorene	ND (2.0)	0.182	ND (2.0)	0.18	ND (2.0)	0.178	ND (1.9)	0.174	***290***
	Indeno(1,2,3-cd)pyrene	ND (1.0)	0.102	ND (1.0)	0.1	ND (0.98)	0.098	ND (0.97)	0.096	***0.19***
	Naphthalene	ND (0.41)	0.2	ND (0.40)	0.2	ND (0.39)	0.196	ND (0.39)	0.194	***1.7***
	Phenanthrene	ND (1.0)	0.26	ND (1.0)	0.26	ND (0.98)	0.26	ND (0.97)	0.26	***170***
	Pyrene	ND (2.0)	0.8	ND (2.0)	0.78	ND (2.0)	0.76	ND (1.9)	0.38	***120***
TAqH[3]			7.6		7.6		7.5		7.1	***15***
Other SVOC	**Nitrobenzene**	0.76 Q	----	ND(0.60) Q	----	ND (0.59)	----	ND (0.58)	----	***1.4***
	Other	None Detected								***Various***

Notes:

Results may be rounded

[1] 18 AAC 70.020; bold values above criteria.

[2] 18 AAC 75.345 Table 3 updated through Sept. 2018; bold values have reporting limits above the cleanup level.

[3] 2 times the individual method detection limits used for summation of non-detect results

ADEC-Alaska Department of Environmental Conservation

EPA-US Environmental Protection Agency

MDL - method detection limit

Q-The result is qualified due to quality control criteria not being met

µg/L-micrograms per liter

ND-not detected (reporting limit)

SVOC - semi-volatile organic compounds

TAH-Total Aromatic Hydrocarbons

TAqH-Total Aqueous Hydrocarbons

VOC - volatile organic compounds

Michael Baker INTERNATIONAL

Ahtna Engineering Services, LLC

(This Page Intentionally Left Blank)

TABLE 2 : ASPHALT SPLP ANALYTICAL RESULTS
INDIAN RIVER ASPHALT PLANT
SITKA NATIONAL HISTORICAL PARK, ALASKA

Sample		EPA Method 1312/8260C SPLP/VOC in mg/kg [1]		EPA Method 1312/8270D-SIM SPLP/PAH in mg/kg [1]
		Bromobenzene	Other VOC	
SITK-18-AS-1		None Detected	None Detected	None Detected
SITK-18-AS-2	Field Duplicate	None Detected	None Detected	None Detected
SITK-18-AS-3		None Detected	None Detected	None Detected
SITK-18-TB-AS-1	Trip Blank[3]	2.0	None Detected	--------
ADEC Cleanup Level MTG [2]		***0.36***	***Various***	***Various***

Notes:

[1] Only detected analytes are listed.

[2] 18 AAC 75.341, Tables B1 and B2, under 40-inch rainfall zone, migration to groundwater (MTG)

[3] Not extracted using SPLP

ADEC - Alaska Department of Environmental Conservation
AK - Alaska
EPA - US Environmental Protection Agency
mg/kg - milligrams per kilogram
ND - Not detected at detection limit shown.
PAH - polycyclic aromatic hydrocarbons
SIM - selected ion monitoring
SPLP - synthetic precipitation leaching procedure
VOC - volatile organic compounds

Michael Baker INTERNATIONAL

Ahtna Engineering Services, LLC

(This Page Intentionally Left Blank)

Appendices

(This Page Intentionally Left Blank)

Appendix A – Photographic Log

Photo 1 – View up bluff from Indian River; view to north.
Date: 8/23/18. Time: 1329.

Photo 2 – View east up Indian River; view to west.
Date: 8/23/18. Time: 1623.

Photo 3 – Asphalt piece lying in gravel.
Date: 8/23/18. Time: 1319.

Photo 4 – Collecting asphalt sample for SPLP-VOC/SVOC analysis.
Date: 8/23/18. Time: 1342.

Photo 5 – Collecting pore water sample PW-1 at low tide; view to west. Note trailers from adjacent trailer park in background.
Date: 8/23/18. Time: 1448.

Photo 6 – Surveying pore water location PW-1 at mouth of the Indian River; view to west.
Date: 8/23/18. Time: 1607.

Appendix B – Field Notes

(This Page Intentionally Left Blank)

INDIAN RIVER ASPHALT PLANT

(SITKA)

Rite in the Rain.

ALL-WEATHER

FIELD

№ 353N

20297.011 NPS

MADE IN TACOMA
SINCE 1916
Rite in the Rain®
DEFYING MOTHER NATURE

Name AHTNA Engineering Services, LLC

Address 110 W. 38th Ave, Suite 200A

Phone 907-646-2969

Email

Projects Indian River Asphalt Plant

RiteintheRain.com

CONTENTS

PAGE	REFERENCE	DATE

INDIAN RIVER ASPHALT PLANT, SITKA

60° A. FITZGERALD 1

Mostly Cloudy August 23, 2018

0630	A. Fitzgerald arrives at Anchorage airport to check in to flight. Meet B Hubner (NPS Engineer) at the airport.
1045	Arrive in Sitka. Field team met by Brennen Carter, NPS Sitka Chief of Resources, who brought field team to site then to NPS headquarter building to put gel ice in freezer.
1230	Field team arrives on site. A. Fitzgerald conducts daily safety meeting. Field team conducts site walk to see where the asphalt and pore water samples will be collected. Mobilize gear and equipment to the beach area.
1315	Set up for asphalt collection at location 1.
1330	Collect asphalt sample SITK-18-AS-1, and duplicate

8/25/18

Sample SITK-18-AS-2 @ 1345.

1355 Set up for asphalt collection at Location 2.

1400 Collect asphalt sample SITK-18-AS-3.
Note: The top layer of asphalt was scraped off to avoid other contaminants (if any).

1420 Set up for pore water sampling at PW-1. Collection slowed to no water and recharge was extremely minimal, so the porewater sampler was relocated closer to the water's edge (the tide was going out at a rapid rate).

1450 Collect pore water sample SITK-18-PW-1, and duplicate sample SITK-18-PW-4 @ 1515.

1540 Set up for pore water sampling at PW-2.

1600 Collect pore water sample SITK-18-PW-2.

1615 Set up for pore water sampling

8/23/18

at PW-3.

1630 Collect pore water sample SITK-18-PW-3.
NOTE: All reusable equipment was decontaminated with Alconox + DI water between each sampling location.

1645 Pack up gear, decon and pack equipment, and carry back to NPS vehicle.
B. Heubner returns to NPS headquarters to retrieve gel ice from freezer and returns to site.

1730 B Heubner completes collection of survey points. Sampling equipment, gear and sample coolers are loaded into vehicle. Field crew departs project site. A. Fitzgerald will QC and repackage sample coolers at hotel. Samples will be brought back to Anchorage and submitted

8/23/18

by hand to Test America tomorrow.

A. FitzGerald + B. Hubner change commercial flight back to Anchorage, departing at 6AM tomorrow.

END of DAY.

8/23/18

~60° Cloudy

August 24, 2018

0500	Field team arrives at Sitka airport for return flight to Anchorage.
0930	
~~0830~~ AF	Arrive in Anchorage, demob equipment + supplies to Ahtna office, TTT and Ahtna warehouse.
1000	Hand deliver samples to Test America, continue demob.
1200	End of Day.

8/24/18

(This Page Intentionally Left Blank)

Appendix C – Laboratory Report

(This Page Intentionally Left Blank)

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

ANALYTICAL REPORT

TestAmerica Laboratories, Inc.
TestAmerica Seattle
5755 8th Street East
Tacoma, WA 98424
Tel: (253)922-2310

TestAmerica Job ID: 580-79910-1
Client Project/Site: Indian River Asphalt Plant-Sitka

For:
Ahtna Engineering Services LLC
110 W 38th Ave
Ste 200A
Anchorage, Alaska 99503

Attn: Nino Muniz

Kristine D. Allen

Authorized for release by:
9/21/2018 4:50:08 PM
Kristine Allen, Manager of Project Management
(253)248-4970
kristine.allen@testamericainc.com

Designee for

Elaine Walker, Project Manager II
(253)248-4972
elaine.walker@testamericainc.com

This report has been electronically signed and authorized by the signatory. Electronic signature is intended to be the legally binding equivalent of a traditionally handwritten signature.

Results relate only to the items tested and the sample(s) as received by the laboratory.

Table of Contents

Job ID: 580-79910-1

Laboratory: TestAmerica Seattle

Narrative

Job Narrative
580-79910-1

Comments
No additional comments.

Receipt
The samples were received on 8/25/2018 9:20 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperatures of the 2 coolers at receipt time were 2.9º C and 2.9º C.

Receipt Exceptions

The following samples were improperly preserved in the field: SITK-18-PW-1 (580-79910-5), SITK-18-PW-4 (580-79910-6), SITK-18-PW-2 (580-79910-7) and SITK-18-PW-3 (580-79910-8). The preservative used is not compatible with the analytes requested.

GC/MS VOA
Method(s) 8260C: The continuing calibration verification (CCV) associated with batch 580-283011 recovered outside acceptance criteria, low biased, for Dichlorodifluoromethane. A reporting limit (RL) standard was analyzed, and the target analyte was detected. Since the associated samples were non-detect for this analyte, the data have been reported.

Method(s) 8260C: The minimum response factor (RF) criteria for the continuing calibration verification (CCV) analyzed in batch 580-283203 was outside criteria for the following analyte(s): Chloroethane and 2-Butanone. As indicated in the reference method, sample analysis may proceed; however, any detection or non-detection for the affected analyte(s) is considered estimated.

Method(s) 8260C: The CCV for analytical batch 580-283203 recovered outside control limits for the following analyte(s): Acetone. Acetone has been identified as a poor performing analyte when analyzed using this method; therefore, re-extraction/re-analysis was not performed. These results have been reported and qualified.

Method(s) 8260C: The following analyte(s) recovered outside control limits for the LCS associated with preparation batch 580-283436 and analytical batch 580-283529: 1,3-Dichloropropane. This is not indicative of a systematic control problem because these were random marginal exceedances. Qualified results have been reported.

Method(s) 8260C: The continuing calibration verification (CCV) associated with batch 580-283529 recovered outside acceptance criteria, low biased, for 1,2,3-Trichloropropane, 2-Hexanone, 1,2,4-Trichlorobenzene, 1,2,3-Trichlorobenzene, 1,1,2,2-Tetrachloroethane, Naphthalene, Dichlorodifluoromethane, Chloromethane, Bromoform, 1,2-Dibromo-3-Chloropropane, 2-Butanone and Methyl tert-butyl ether. A reporting limit (RL) standard was analyzed, and the target analyte was detected. Since the associated samples were non-detect for this analyte, the data have been reported.

Method(s) 8260C: The minimum response factor (RF) criteria for the continuing calibration verification (CCV) analyzed in batch 580-283529 was outside criteria for the following analyte(s): 1,3-Dichlorobenzene. As indicated in the reference method, sample analysis may proceed; however, any detection or non-detection for the affected analyte(s) is considered estimated.

Method(s) 8260C: The container used for reanalysis of the following sample contained headspace: SITK-18-AS-2 (580-79910-3) The method used for analysis requires that the sample does not contain headspace. The septa had failed causing the vial to lose integrity.

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

GC/MS Semi VOA
Method(s) 8270D: The continuing calibration verification (CCV) associated with batch 580-283155 recovered above the upper control limit for 2,4-Dinitrophenol and Di-n-octyl phthalate. The samples associated with this CCV were non-detects for the affected analytes; therefore, the data have been reported. The following samples are impacted: SITK-18-PW-1 (580-79910-5), SITK-18-PW-4 (580-79910-6), SITK-18-PW-2 (580-79910-7), SITK-18-PW-3 (580-79910-8) and (CCVIS 580-283155/3).

Method(s) 8270D: The minimum response factor (RF) criteria for the continuing calibration verification (CCV) analyzed in batch 580-283155 was outside criteria for the following analyte: N-Nitrosodi-n-propylamine. As indicated in the reference method, sample

Job ID: 580-79910-1 (Continued)

Laboratory: TestAmerica Seattle (Continued)

analysis may proceed; however, any detection or non-detection for the affected analyte(s) is considered estimated.

Method(s) 8270D: The following analytes recovered outside control limits for the LCS/LCSD associated with preparation batch 580-282779 and analytical batch 580-283155: Benzyl alcohol (LCS only), 4-Chloroaniline, Hexachlorocyclopentadiene, and Bis(2-ethylhexyl) phthalate (LCSD only). This is not indicative of a systematic control problem because these were random marginal exceedances. Qualified results have been reported.

Method(s) 8270D: The RPD of the laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) for batch preparation batch 580-282779 and analytical batch 580-283155 recovered outside control limits for the following analyte: 4-Chloroaniline.

Method(s) 8270D: The continuing calibration verification (CCV) associated with batch 580-283944 recovered outside acceptance criteria, low biased, for 4-Nitroaniline, 3,3'-Dichlorobenzidine, Benzo[b]fluoranthene, Benzo[k]fluoranthene, Indeno[1,2,3-cd]pyrene, Dibenz(a,h)anthracene and Benzo[g,h,i]perylene. A standard was analyzed at or below the reporting limit (RL), and the target analytes were detected. Since the associated samples were non-detect for this analyte, the data have been reported.

Method(s) 8270D: The following analyte(s) recovered outside control limits for the LCS/LCSD associated with preparation batch 580-283880 and analytical batch 580-283944: 1,3-Dichlorobenzene, Bis(2-chloroethyl)ether (LCS only), 2,4-Dinitrophenol (LCS only), and Butyl benzyl phthalate (LCS only). These analytes were outside the Marginal Exceedance Limits. However, since these samples are SPLP samples and not all analytes recover well in the leachate: the data is qualified and reported.

Method(s) 8270D: The RPD of the laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) for batch preparation batch 580-283880 and analytical batch 580-283944 recovered outside control limits for the following analytes: Phenol, Pyrene, Fluoranthene, Bis(2-chloroethyl)ether, 1,2,4-Trichlorobenzene, Isophorone, Acenaphthene, Benzo[g,h,i]perylene, Bis(2-chloroethoxy)methane, 2-Chlorophenol, and Dibenz(a,h)anthracene.

Method(s) 8270D: Internal standard (ISTD) response for the following method blank was outside of acceptance limits: (MB 580-283435/1-B). Since a low internal standard yields high bias on detections for the affected analytes, and all the affected analytes were ND in the method blank, the data is qualified and reported.

Method(s) 8270D: The continuing calibration verification (CCV) associated with batch 580-283944 recovered above the upper control limit for 4-Nitrophenol, Di-n-butyl phthalate, 2,4,6-Trichlorophenol, 2-Nitroaniline, Hexachloroethane, 3-Nitroaniline, Butyl benzyl phthalate, Pentachlorophenol, 2,6-Dinitrotoluene, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 2,4-Dinitrotoluene. The samples associated with this CCV were non-detects for the affected analytes; therefore, the data have been reported.

Method(s) 8270D: Six surrogates are used for this analysis. The laboratory's SOP allows three of these surrogates to be outside acceptance criteria without performing re-extraction/re-analysis. The following samples contained an allowable number of surrogate compounds outside limits: SITK-18-AS-2 (580-79910-3), SITK-18-AS-3 (580-79910-4) and (580-79910-A-1-D MS). These results have been reported and qualified.

Method(s) 8270D: The following CCV recovered outside acceptance criteria for %D for surrogate 2-Fluorophenol (Surr). Since the %Rec is within the acceptance criteria for the surrogate in the CCV and associated samples, the data have been reported. SITK-18-AS-1 (580-79910-1), SITK-18-AS-2 (580-79910-3), SITK-18-AS-3 (580-79910-4) and (CCVIS 580-283944/3)

Method(s) 8270D: The minimum response factor (RF) criteria for the continuing calibration verification (CCV) analyzed in batch 580-283944 was outside criteria for the following analyte(s): N-Nitrosodi-n-propylamine. As indicated in the reference method, sample analysis may proceed; however, any detection or non-detection for the affected analyte(s) is considered estimated.

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

General Chemistry
No analytical or quality issues were noted, other than those described in the Definitions/Glossary page.

Organic Prep
Method(s) 3520C: The samples arrived preserved in HCl, when they should be unpreserved. pH was tested before extraction to ensure a pH of 2 had been achieved. If the samples needed to be acidified further, sulfuric acid would be used.

Job ID: 580-79910-1 (Continued)

Laboratory: TestAmerica Seattle (Continued)

SITK-18-PW-1 (580-79910-5), SITK-18-PW-4 (580-79910-6), SITK-18-PW-2 (580-79910-7) and SITK-18-PW-3 (580-79910-8)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Qualifiers

GC/MS VOA

Qualifier	Qualifier Description
*	LCS or LCSD is outside acceptance limits.
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.

GC/MS Semi VOA

Qualifier	Qualifier Description
*	RPD of the LCS and LCSD exceeds the control limits
*	LCS or LCSD is outside acceptance limits.
F1	MS and/or MSD Recovery is outside acceptance limits.
X	Surrogate is outside control limits
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
*	ISTD response or retention time outside acceptable limits

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
PQL	Practical Quantitation Limit
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-1
Date Collected: 08/23/18 13:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-1
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		200	18	ug/L			09/08/18 11:30	100
1,1,1-Trichloroethane	ND		300	14	ug/L			09/08/18 11:30	100
1,1,2,2-Tetrachloroethane	ND		300	52	ug/L			09/08/18 11:30	100
1,1,2-Trichloroethane	ND		100	24	ug/L			09/18/18 15:39	100
1,1-Dichloroethane	ND		200	22	ug/L			09/08/18 11:30	100
1,1-Dichloroethene	ND		400	78	ug/L			09/08/18 11:30	100
1,1-Dichloropropene	ND		300	29	ug/L			09/08/18 11:30	100
1,2,3-Trichlorobenzene	ND		500	46	ug/L			09/08/18 11:30	100
1,2,3-Trichloropropane	ND		200	41	ug/L			09/08/18 11:30	100
1,2,4-Trichlorobenzene	ND		200	33	ug/L			09/08/18 11:30	100
1,2,4-Trimethylbenzene	ND		300	61	ug/L			09/08/18 11:30	100
1,2-Dibromo-3-Chloropropane	ND		1000	180	ug/L			09/08/18 11:30	100
1,2-Dibromoethane	ND		200	40	ug/L			09/18/18 15:39	100
1,2-Dichlorobenzene	ND		200	46	ug/L			09/08/18 11:30	100
1,2-Dichloroethane	ND		200	53	ug/L			09/08/18 11:30	100
1,2-Dichloropropane	ND		100	18	ug/L			09/08/18 11:30	100
1,3,5-Trimethylbenzene	ND		300	55	ug/L			09/08/18 11:30	100
1,3-Dichlorobenzene	ND		200	18	ug/L			09/08/18 11:30	100
1,3-Dichloropropane	ND	*	200	35	ug/L			09/08/18 11:30	100
1,4-Dichlorobenzene	ND		400	98	ug/L			09/08/18 11:30	100
2,2-Dichloropropane	ND		300	32	ug/L			09/08/18 11:30	100
2-Butanone	ND		2000	470	ug/L			09/08/18 11:30	100
2-Chlorotoluene	ND		300	51	ug/L			09/08/18 11:30	100
2-Hexanone	ND		2000	400	ug/L			09/08/18 11:30	100
4-Chlorotoluene	ND		200	51	ug/L			09/08/18 11:30	100
4-Isopropyltoluene	ND		300	28	ug/L			09/08/18 11:30	100
4-Methyl-2-pentanone	ND		1500	250	ug/L			09/18/18 15:39	100
Acetone	ND		5000	780	ug/L			09/08/18 11:30	100
Benzene	ND		300	53	ug/L			09/08/18 11:30	100
Bromobenzene	ND		200	18	ug/L			09/08/18 11:30	100
Bromochloromethane	ND		200	29	ug/L			09/08/18 11:30	100
Bromodichloromethane	ND		200	14	ug/L			09/08/18 11:30	100
Bromoform	ND		300	56	ug/L			09/08/18 11:30	100
Bromomethane	ND		600	110	ug/L			09/08/18 11:30	100
Carbon disulfide	ND		300	53	ug/L			09/08/18 11:30	100
Carbon tetrachloride	ND		300	30	ug/L			09/08/18 11:30	100
Chlorobenzene	ND		200	44	ug/L			09/08/18 11:30	100
Chloroethane	ND		500	110	ug/L			09/08/18 11:30	100
Chloroform	ND		500	50	ug/L			09/08/18 11:30	100
Chloromethane	ND		2000	540	ug/L			09/08/18 11:30	100
cis-1,2-Dichloroethene	ND		300	69	ug/L			09/08/18 11:30	100
cis-1,3-Dichloropropene	ND		100	20	ug/L			09/08/18 11:30	100
Dibromochloromethane	ND		100	20	ug/L			09/08/18 11:30	100
Dibromomethane	ND		200	34	ug/L			09/18/18 15:39	100
Dichlorodifluoromethane	ND		1000	230	ug/L			09/08/18 11:30	100
Ethylbenzene	ND		300	50	ug/L			09/08/18 11:30	100
Hexachlorobutadiene	ND		600	79	ug/L			09/08/18 11:30	100
Isopropylbenzene	ND		200	51	ug/L			09/08/18 11:30	100
Methyl tert-butyl ether	ND		200	44	ug/L			09/08/18 11:30	100

Client Sample ID: SITK-18-AS-1
Date Collected: 08/23/18 13:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-1
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		500	140	ug/L			09/08/18 11:30	100
m-Xylene & p-Xylene	ND		300	75	ug/L			09/08/18 11:30	100
Naphthalene	ND		400	93	ug/L			09/08/18 11:30	100
n-Butylbenzene	ND		300	44	ug/L			09/08/18 11:30	100
N-Propylbenzene	ND		300	50	ug/L			09/08/18 11:30	100
o-Xylene	ND		200	15	ug/L			09/08/18 11:30	100
sec-Butylbenzene	ND		300	49	ug/L			09/08/18 11:30	100
Styrene	ND		500	51	ug/L			09/08/18 11:30	100
t-Butylbenzene	ND		300	58	ug/L			09/08/18 11:30	100
Tetrachloroethene	ND		300	41	ug/L			09/08/18 11:30	100
Toluene	ND		200	39	ug/L			09/08/18 11:30	100
trans-1,2-Dichloroethene	ND		300	39	ug/L			09/08/18 11:30	100
trans-1,3-Dichloropropene	ND		100	16	ug/L			09/08/18 11:30	100
Trichloroethene	ND		300	85	ug/L			09/08/18 11:30	100
Trichlorofluoromethane	ND		300	63	ug/L			09/08/18 11:30	100
Vinyl chloride	ND		100	22	ug/L			09/08/18 11:30	100

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*87*		*80 - 126*		*09/08/18 11:30*	*100*
1,2-Dichloroethane-d4 (Surr)	*99*		*80 - 126*		*09/18/18 15:39*	*100*
4-Bromofluorobenzene (Surr)	*94*		*80 - 125*		*09/08/18 11:30*	*100*
4-Bromofluorobenzene (Surr)	*101*		*80 - 125*		*09/18/18 15:39*	*100*
Dibromofluoromethane (Surr)	*95*		*77 - 120*		*09/08/18 11:30*	*100*
Dibromofluoromethane (Surr)	*100*		*77 - 120*		*09/18/18 15:39*	*100*
Toluene-d8 (Surr)	*104*		*80 - 122*		*09/08/18 11:30*	*100*
Toluene-d8 (Surr)	*104*		*80 - 122*		*09/18/18 15:39*	*100*
Trifluorotoluene (Surr)	*105*		*80 - 120*		*09/08/18 11:30*	*100*
Trifluorotoluene (Surr)	*98*		*80 - 120*		*09/18/18 15:39*	*100*

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Phenol	ND	*	20	1.8	ug/L		09/13/18 10:05	09/13/18 21:40	1
Bis(2-chloroethyl)ether	ND	*	3.0	0.15	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Chlorophenol	ND	*	5.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
1,3-Dichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
1,4-Dichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzyl alcohol	ND		15	0.75	ug/L		09/13/18 10:05	09/13/18 21:40	1
1,2-Dichlorobenzene	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Methylphenol	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
3 & 4 Methylphenol	ND		4.0	0.15	ug/L		09/13/18 10:05	09/13/18 21:40	1
N-Nitrosodi-n-propylamine	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
Hexachloroethane	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
Nitrobenzene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Isophorone	ND	*	2.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Nitrophenol	ND		5.0	0.75	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4-Dimethylphenol	ND		20	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzoic acid	ND		20	3.5	ug/L		09/13/18 10:05	09/13/18 21:40	1
Bis(2-chloroethoxy)methane	ND	*	3.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4-Dichlorophenol	ND	*	20	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
1,2,4-Trichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1

Client Sample ID: SITK-18-AS-1
Date Collected: 08/23/18 13:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-1
Matrix: Solid

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Naphthalene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Chloroaniline	ND		50	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
Hexachlorobutadiene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Chloro-3-methylphenol	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Methylnaphthalene	ND		2.0	0.15	ug/L		09/13/18 10:05	09/13/18 21:40	1
Hexachlorocyclopentadiene	ND		25	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4,6-Trichlorophenol	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4,5-Trichlorophenol	ND		2.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Chloronaphthalene	ND	F1	5.0	0.15	ug/L		09/13/18 10:05	09/13/18 21:40	1
2-Nitroaniline	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
Dimethyl phthalate	ND	F1	3.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
Acenaphthylene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,6-Dinitrotoluene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
3-Nitroaniline	ND		15	0.15	ug/L		09/13/18 10:05	09/13/18 21:40	1
Acenaphthene	ND	*	2.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4-Dinitrophenol	ND		25	1.4	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Nitrophenol	ND	F1	75	2.6	ug/L		09/13/18 10:05	09/13/18 21:40	1
Dibenzofuran	ND		2.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
2,4-Dinitrotoluene	ND		5.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
Diethyl phthalate	ND		60	2.8	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Chlorophenyl phenyl ether	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
Fluorene	ND		10	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Nitroaniline	ND		10	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
4,6-Dinitro-2-methylphenol	ND		25	1.3	ug/L		09/13/18 10:05	09/13/18 21:40	1
N-Nitrosodiphenylamine	ND		75	16	ug/L		09/13/18 10:05	09/13/18 21:40	1
4-Bromophenyl phenyl ether	ND		3.0	0.10	ug/L		09/13/18 10:05	09/13/18 21:40	1
Hexachlorobenzene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Pentachlorophenol	ND		50	1.3	ug/L		09/13/18 10:05	09/13/18 21:40	1
Phenanthrene	ND		5.0	1.2	ug/L		09/13/18 10:05	09/13/18 21:40	1
Anthracene	ND		75	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
Di-n-butyl phthalate	ND		15	2.2	ug/L		09/13/18 10:05	09/13/18 21:40	1
Fluoranthene	ND	*	15	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
Pyrene	ND	*	10	0.55	ug/L		09/13/18 10:05	09/13/18 21:40	1
Butyl benzyl phthalate	ND	*	50	4.7	ug/L		09/13/18 10:05	09/13/18 21:40	1
3,3'-Dichlorobenzidine	ND		75	0.95	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzo[a]anthracene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
Chrysene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Bis(2-ethylhexyl) phthalate	ND		75	13	ug/L		09/13/18 10:05	09/13/18 21:40	1
Di-n-octyl phthalate	ND		5.0	0.65	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzo[a]pyrene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Indeno[1,2,3-cd]pyrene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1
Dibenz(a,h)anthracene	ND	*	3.0	0.35	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzo[g,h,i]perylene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Carbazole	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 21:40	1
1-Methylnaphthalene	ND		5.0	0.10	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzo[b]fluoranthene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 21:40	1
Benzo[k]fluoranthene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 21:40	1
bis(chloroisopropyl) ether	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 21:40	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-1
Date Collected: 08/23/18 13:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-1
Matrix: Solid

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
2-Fluorophenol (Surr)	54		20 - 147	09/13/18 10:05	09/13/18 21:40	1
Phenol-d5 (Surr)	55		21 - 135	09/13/18 10:05	09/13/18 21:40	1
Nitrobenzene-d5 (Surr)	87		60 - 120	09/13/18 10:05	09/13/18 21:40	1
2-Fluorobiphenyl	68		63 - 120	09/13/18 10:05	09/13/18 21:40	1
2,4,6-Tribromophenol (Surr)	91		28 - 131	09/13/18 10:05	09/13/18 21:40	1
Terphenyl-d14 (Surr)	87		66 - 120	09/13/18 10:05	09/13/18 21:40	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-TB-AS-1
Date Collected: 08/23/18 08:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-2
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		40	11	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1,1-Trichloroethane	ND		40	9.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1,2,2-Tetrachloroethane	ND		20	7.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1,2-Trichloroethane	ND		20	7.4	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1-Dichloroethane	ND		40	4.2	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1-Dichloroethene	ND		40	12	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,1-Dichloropropene	ND		40	5.3	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2,3-Trichlorobenzene	ND		150	32	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2,3-Trichloropropane	ND		40	12	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2,4-Trichlorobenzene	ND		60	15	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2,4-Trimethylbenzene	ND		40	14	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2-Dibromo-3-Chloropropane	ND		250	40	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2-Dibromoethane	ND		20	3.8	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2-Dichlorobenzene	ND		40	8.7	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2-Dichloroethane	ND		20	5.5	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,2-Dichloropropane	ND		20	6.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,3,5-Trimethylbenzene	ND		40	7.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,3-Dichlorobenzene	ND		60	13	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,3-Dichloropropane	ND		60	14	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
1,4-Dichlorobenzene	ND		60	11	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
2,2-Dichloropropane	ND		40	12	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
2-Butanone	ND		600	190	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
2-Chlorotoluene	ND		40	8.8	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
2-Hexanone	ND		100	36	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
4-Chlorotoluene	ND		40	9.8	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
4-Isopropyltoluene	ND		40	10	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
4-Methyl-2-pentanone	ND		400	81	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Acetone	ND		800	170	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Benzene	ND		30	7.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Bromobenzene	**2000**		100	17	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Bromochloromethane	ND		40	6.2	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Bromodichloromethane	ND		60	13	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Bromoform	ND		200	26	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Bromomethane	ND		200	13	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Carbon disulfide	ND		60	12	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Carbon tetrachloride	ND		20	3.8	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Chlorobenzene	ND		40	9.8	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Chloroethane	ND		400	54	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Chloroform	ND		40	4.2	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Chloromethane	ND		100	10	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
cis-1,2-Dichloroethene	ND		60	13	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
cis-1,3-Dichloropropene	ND		20	4.0	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Dibromochloromethane	ND		40	11	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Dibromomethane	ND		60	7.4	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Dichlorodifluoromethane	ND		200	46	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Ethylbenzene	ND		40	9.1	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Hexachlorobutadiene	ND		150	33	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Isopropylbenzene	ND		40	8.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Methyl tert-butyl ether	ND		40	6.0	ug/Kg		09/04/18 18:30	09/05/18 04:23	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-TB-AS-1
Date Collected: 08/23/18 08:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-2
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		250	65	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
m-Xylene & p-Xylene	ND		200	15	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Naphthalene	ND		100	28	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
n-Butylbenzene	ND		150	25	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
N-Propylbenzene	ND		40	6.9	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
o-Xylene	ND		60	13	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
sec-Butylbenzene	ND		40	8.6	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Styrene	ND		40	6.1	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
t-Butylbenzene	ND		40	7.7	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Tetrachloroethene	ND		40	5.3	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Toluene	ND		150	14	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
trans-1,2-Dichloroethene	ND		60	15	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
trans-1,3-Dichloropropene	ND		40	7.0	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Trichloroethene	ND		60	22	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Trichlorofluoromethane	ND		200	11	ug/Kg		09/04/18 18:30	09/05/18 04:23	1
Vinyl chloride	ND		150	26	ug/Kg		09/04/18 18:30	09/05/18 04:23	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*94*		*80 - 121*	*09/04/18 18:30*	*09/05/18 04:23*	*1*
4-Bromofluorobenzene (Surr)	*117*		*80 - 120*	*09/04/18 18:30*	*09/05/18 04:23*	*1*
Dibromofluoromethane (Surr)	*99*		*80 - 120*	*09/04/18 18:30*	*09/05/18 04:23*	*1*
Toluene-d8 (Surr)	*99*		*80 - 120*	*09/04/18 18:30*	*09/05/18 04:23*	*1*
Trifluorotoluene (Surr)	*112*		*80 - 120*	*09/04/18 18:30*	*09/05/18 04:23*	*1*

Client Sample ID: SITK-18-AS-2
Date Collected: 08/23/18 13:45
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-3
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		200	18	ug/L			09/08/18 11:56	100
1,1,1-Trichloroethane	ND		300	14	ug/L			09/08/18 11:56	100
1,1,2,2-Tetrachloroethane	ND		300	52	ug/L			09/08/18 11:56	100
1,1,2-Trichloroethane	ND		100	24	ug/L			09/18/18 16:04	100
1,1-Dichloroethane	ND		200	22	ug/L			09/08/18 11:56	100
1,1-Dichloroethene	ND		400	78	ug/L			09/08/18 11:56	100
1,1-Dichloropropene	ND		300	29	ug/L			09/08/18 11:56	100
1,2,3-Trichlorobenzene	ND		500	46	ug/L			09/08/18 11:56	100
1,2,3-Trichloropropane	ND		200	41	ug/L			09/08/18 11:56	100
1,2,4-Trichlorobenzene	ND		200	33	ug/L			09/08/18 11:56	100
1,2,4-Trimethylbenzene	ND		300	61	ug/L			09/08/18 11:56	100
1,2-Dibromo-3-Chloropropane	ND		1000	180	ug/L			09/08/18 11:56	100
1,2-Dibromoethane	ND		200	40	ug/L			09/18/18 16:04	100
1,2-Dichlorobenzene	ND		200	46	ug/L			09/08/18 11:56	100
1,2-Dichloroethane	ND		200	53	ug/L			09/08/18 11:56	100
1,2-Dichloropropane	ND		100	18	ug/L			09/08/18 11:56	100
1,3,5-Trimethylbenzene	ND		300	55	ug/L			09/08/18 11:56	100
1,3-Dichlorobenzene	ND		200	18	ug/L			09/08/18 11:56	100
1,3-Dichloropropane	ND	*	200	35	ug/L			09/08/18 11:56	100
1,4-Dichlorobenzene	ND		400	98	ug/L			09/08/18 11:56	100
2,2-Dichloropropane	ND		300	32	ug/L			09/08/18 11:56	100
2-Butanone	ND		2000	470	ug/L			09/08/18 11:56	100
2-Chlorotoluene	ND		300	51	ug/L			09/08/18 11:56	100
2-Hexanone	ND		2000	400	ug/L			09/08/18 11:56	100
4-Chlorotoluene	ND		200	51	ug/L			09/08/18 11:56	100
4-Isopropyltoluene	ND		300	28	ug/L			09/08/18 11:56	100
4-Methyl-2-pentanone	ND		1500	250	ug/L			09/18/18 16:04	100
Acetone	ND		5000	780	ug/L			09/08/18 11:56	100
Benzene	ND		300	53	ug/L			09/08/18 11:56	100
Bromobenzene	ND		200	18	ug/L			09/08/18 11:56	100
Bromochloromethane	ND		200	29	ug/L			09/08/18 11:56	100
Bromodichloromethane	ND		200	14	ug/L			09/08/18 11:56	100
Bromoform	ND		300	56	ug/L			09/08/18 11:56	100
Bromomethane	ND		600	110	ug/L			09/08/18 11:56	100
Carbon disulfide	ND		300	53	ug/L			09/08/18 11:56	100
Carbon tetrachloride	ND		300	30	ug/L			09/08/18 11:56	100
Chlorobenzene	ND		200	44	ug/L			09/08/18 11:56	100
Chloroethane	ND		500	110	ug/L			09/08/18 11:56	100
Chloroform	ND		500	50	ug/L			09/08/18 11:56	100
Chloromethane	ND		2000	540	ug/L			09/08/18 11:56	100
cis-1,2-Dichloroethene	ND		300	69	ug/L			09/08/18 11:56	100
cis-1,3-Dichloropropene	ND		100	20	ug/L			09/08/18 11:56	100
Dibromochloromethane	ND		100	20	ug/L			09/08/18 11:56	100
Dibromomethane	ND		200	34	ug/L			09/18/18 16:04	100
Dichlorodifluoromethane	ND		1000	230	ug/L			09/08/18 11:56	100
Ethylbenzene	ND		300	50	ug/L			09/08/18 11:56	100
Hexachlorobutadiene	ND		600	79	ug/L			09/08/18 11:56	100
Isopropylbenzene	ND		200	51	ug/L			09/08/18 11:56	100
Methyl tert-butyl ether	ND		200	44	ug/L			09/08/18 11:56	100

Client Sample ID: SITK-18-AS-2
Date Collected: 08/23/18 13:45
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-3
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		500	140	ug/L			09/08/18 11:56	100
m-Xylene & p-Xylene	ND		300	75	ug/L			09/08/18 11:56	100
Naphthalene	ND		400	93	ug/L			09/08/18 11:56	100
n-Butylbenzene	ND		300	44	ug/L			09/08/18 11:56	100
N-Propylbenzene	ND		300	50	ug/L			09/08/18 11:56	100
o-Xylene	ND		200	15	ug/L			09/08/18 11:56	100
sec-Butylbenzene	ND		300	49	ug/L			09/08/18 11:56	100
Styrene	ND		500	51	ug/L			09/08/18 11:56	100
t-Butylbenzene	ND		300	58	ug/L			09/08/18 11:56	100
Tetrachloroethene	ND		300	41	ug/L			09/08/18 11:56	100
Toluene	ND		200	39	ug/L			09/08/18 11:56	100
trans-1,2-Dichloroethene	ND		300	39	ug/L			09/08/18 11:56	100
trans-1,3-Dichloropropene	ND		100	16	ug/L			09/08/18 11:56	100
Trichloroethene	ND		300	85	ug/L			09/08/18 11:56	100
Trichlorofluoromethane	ND		300	63	ug/L			09/08/18 11:56	100
Vinyl chloride	ND		100	22	ug/L			09/08/18 11:56	100

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*91*		*80 - 126*		*09/08/18 11:56*	*100*
1,2-Dichloroethane-d4 (Surr)	*105*		*80 - 126*		*09/18/18 16:04*	*100*
4-Bromofluorobenzene (Surr)	*95*		*80 - 125*		*09/08/18 11:56*	*100*
4-Bromofluorobenzene (Surr)	*102*		*80 - 125*		*09/18/18 16:04*	*100*
Dibromofluoromethane (Surr)	*98*		*77 - 120*		*09/08/18 11:56*	*100*
Dibromofluoromethane (Surr)	*103*		*77 - 120*		*09/18/18 16:04*	*100*
Toluene-d8 (Surr)	*104*		*80 - 122*		*09/08/18 11:56*	*100*
Toluene-d8 (Surr)	*103*		*80 - 122*		*09/18/18 16:04*	*100*
Trifluorotoluene (Surr)	*104*		*80 - 120*		*09/08/18 11:56*	*100*
Trifluorotoluene (Surr)	*102*		*80 - 120*		*09/18/18 16:04*	*100*

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Phenol	ND	*	20	1.8	ug/L		09/13/18 10:05	09/13/18 22:31	1
Bis(2-chloroethyl)ether	ND	*	3.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Chlorophenol	ND	*	5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
1,3-Dichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
1,4-Dichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzyl alcohol	ND		15	0.75	ug/L		09/13/18 10:05	09/13/18 22:31	1
1,2-Dichlorobenzene	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Methylphenol	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
3 & 4 Methylphenol	ND		4.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:31	1
N-Nitrosodi-n-propylamine	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
Hexachloroethane	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
Nitrobenzene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Isophorone	ND	*	2.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Nitrophenol	ND		5.0	0.75	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4-Dimethylphenol	ND		20	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzoic acid	ND		20	3.5	ug/L		09/13/18 10:05	09/13/18 22:31	1
Bis(2-chloroethoxy)methane	ND	*	3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4-Dichlorophenol	ND	*	20	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
1,2,4-Trichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-2
Date Collected: 08/23/18 13:45
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-3
Matrix: Solid

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Naphthalene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Chloroaniline	ND		50	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
Hexachlorobutadiene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Chloro-3-methylphenol	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Methylnaphthalene	ND		2.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:31	1
Hexachlorocyclopentadiene	ND		25	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4,6-Trichlorophenol	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4,5-Trichlorophenol	ND		2.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Chloronaphthalene	ND		5.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:31	1
2-Nitroaniline	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
Dimethyl phthalate	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
Acenaphthylene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,6-Dinitrotoluene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
3-Nitroaniline	ND		15	0.15	ug/L		09/13/18 10:05	09/13/18 22:31	1
Acenaphthene	ND	*	2.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4-Dinitrophenol	ND		25	1.4	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Nitrophenol	ND		75	2.6	ug/L		09/13/18 10:05	09/13/18 22:31	1
Dibenzofuran	ND		2.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
2,4-Dinitrotoluene	ND		5.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
Diethyl phthalate	ND		60	2.8	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Chlorophenyl phenyl ether	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
Fluorene	ND		10	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Nitroaniline	ND		10	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
4,6-Dinitro-2-methylphenol	ND		25	1.3	ug/L		09/13/18 10:05	09/13/18 22:31	1
N-Nitrosodiphenylamine	ND		75	16	ug/L		09/13/18 10:05	09/13/18 22:31	1
4-Bromophenyl phenyl ether	ND		3.0	0.10	ug/L		09/13/18 10:05	09/13/18 22:31	1
Hexachlorobenzene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Pentachlorophenol	ND		50	1.3	ug/L		09/13/18 10:05	09/13/18 22:31	1
Phenanthrene	ND		5.0	1.2	ug/L		09/13/18 10:05	09/13/18 22:31	1
Anthracene	ND		75	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
Di-n-butyl phthalate	ND		15	2.2	ug/L		09/13/18 10:05	09/13/18 22:31	1
Fluoranthene	ND	*	15	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
Pyrene	ND	*	10	0.55	ug/L		09/13/18 10:05	09/13/18 22:31	1
Butyl benzyl phthalate	ND	*	50	4.7	ug/L		09/13/18 10:05	09/13/18 22:31	1
3,3'-Dichlorobenzidine	ND		75	0.95	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzo[a]anthracene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
Chrysene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Bis(2-ethylhexyl) phthalate	ND		75	13	ug/L		09/13/18 10:05	09/13/18 22:31	1
Di-n-octyl phthalate	ND		5.0	0.65	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzo[a]pyrene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Indeno[1,2,3-cd]pyrene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1
Dibenz(a,h)anthracene	ND	*	3.0	0.35	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzo[g,h,i]perylene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Carbazole	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:31	1
1-Methylnaphthalene	ND		5.0	0.10	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzo[b]fluoranthene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:31	1
Benzo[k]fluoranthene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:31	1
bis(chloroisopropyl) ether	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:31	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-2
Date Collected: 08/23/18 13:45
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-3
Matrix: Solid

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
2-Fluorophenol (Surr)	55		20 - 147	09/13/18 10:05	09/13/18 22:31	1
Phenol-d5 (Surr)	44		21 - 135	09/13/18 10:05	09/13/18 22:31	1
Nitrobenzene-d5 (Surr)	64		60 - 120	09/13/18 10:05	09/13/18 22:31	1
2-Fluorobiphenyl	56	X	63 - 120	09/13/18 10:05	09/13/18 22:31	1
2,4,6-Tribromophenol (Surr)	90		28 - 131	09/13/18 10:05	09/13/18 22:31	1
Terphenyl-d14 (Surr)	79		66 - 120	09/13/18 10:05	09/13/18 22:31	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-3
Date Collected: 08/23/18 14:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-4
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		200	18	ug/L			09/08/18 12:22	100
1,1,1-Trichloroethane	ND		300	14	ug/L			09/08/18 12:22	100
1,1,2,2-Tetrachloroethane	ND		300	52	ug/L			09/08/18 12:22	100
1,1,2-Trichloroethane	ND		100	24	ug/L			09/18/18 16:28	100
1,1-Dichloroethane	ND		200	22	ug/L			09/08/18 12:22	100
1,1-Dichloroethene	ND		400	78	ug/L			09/08/18 12:22	100
1,1-Dichloropropene	ND		300	29	ug/L			09/08/18 12:22	100
1,2,3-Trichlorobenzene	ND		500	46	ug/L			09/08/18 12:22	100
1,2,3-Trichloropropane	ND		200	41	ug/L			09/08/18 12:22	100
1,2,4-Trichlorobenzene	ND		200	33	ug/L			09/08/18 12:22	100
1,2,4-Trimethylbenzene	ND		300	61	ug/L			09/08/18 12:22	100
1,2-Dibromo-3-Chloropropane	ND		1000	180	ug/L			09/08/18 12:22	100
1,2-Dibromoethane	ND		200	40	ug/L			09/18/18 16:28	100
1,2-Dichlorobenzene	ND		200	46	ug/L			09/08/18 12:22	100
1,2-Dichloroethane	ND		200	53	ug/L			09/08/18 12:22	100
1,2-Dichloropropane	ND		100	18	ug/L			09/08/18 12:22	100
1,3,5-Trimethylbenzene	ND		300	55	ug/L			09/08/18 12:22	100
1,3-Dichlorobenzene	ND		200	18	ug/L			09/08/18 12:22	100
1,3-Dichloropropane	ND	*	200	35	ug/L			09/08/18 12:22	100
1,4-Dichlorobenzene	ND		400	98	ug/L			09/08/18 12:22	100
2,2-Dichloropropane	ND		300	32	ug/L			09/08/18 12:22	100
2-Butanone	ND		2000	470	ug/L			09/08/18 12:22	100
2-Chlorotoluene	ND		300	51	ug/L			09/08/18 12:22	100
2-Hexanone	ND		2000	400	ug/L			09/08/18 12:22	100
4-Chlorotoluene	ND		200	51	ug/L			09/08/18 12:22	100
4-Isopropyltoluene	ND		300	28	ug/L			09/08/18 12:22	100
4-Methyl-2-pentanone	ND		1500	250	ug/L			09/18/18 16:28	100
Acetone	ND		5000	780	ug/L			09/08/18 12:22	100
Benzene	ND		300	53	ug/L			09/08/18 12:22	100
Bromobenzene	ND		200	18	ug/L			09/08/18 12:22	100
Bromochloromethane	ND		200	29	ug/L			09/08/18 12:22	100
Bromodichloromethane	ND		200	14	ug/L			09/08/18 12:22	100
Bromoform	ND		300	56	ug/L			09/08/18 12:22	100
Bromomethane	ND		600	110	ug/L			09/08/18 12:22	100
Carbon disulfide	ND		300	53	ug/L			09/08/18 12:22	100
Carbon tetrachloride	ND		300	30	ug/L			09/08/18 12:22	100
Chlorobenzene	ND		200	44	ug/L			09/08/18 12:22	100
Chloroethane	ND		500	110	ug/L			09/08/18 12:22	100
Chloroform	ND		500	50	ug/L			09/08/18 12:22	100
Chloromethane	ND		2000	540	ug/L			09/08/18 12:22	100
cis-1,2-Dichloroethene	ND		300	69	ug/L			09/08/18 12:22	100
cis-1,3-Dichloropropene	ND		100	20	ug/L			09/08/18 12:22	100
Dibromochloromethane	ND		100	20	ug/L			09/08/18 12:22	100
Dibromomethane	ND		200	34	ug/L			09/18/18 16:28	100
Dichlorodifluoromethane	ND		1000	230	ug/L			09/08/18 12:22	100
Ethylbenzene	ND		300	50	ug/L			09/08/18 12:22	100
Hexachlorobutadiene	ND		600	79	ug/L			09/08/18 12:22	100
Isopropylbenzene	ND		200	51	ug/L			09/08/18 12:22	100
Methyl tert-butyl ether	ND		200	44	ug/L			09/08/18 12:22	100

Client Sample ID: SITK-18-AS-3
Date Collected: 08/23/18 14:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-4
Matrix: Solid

Method: 8260C - Volatile Organic Compounds by GC/MS - SPLP (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		500	140	ug/L			09/08/18 12:22	100
m-Xylene & p-Xylene	ND		300	75	ug/L			09/08/18 12:22	100
Naphthalene	ND		400	93	ug/L			09/08/18 12:22	100
n-Butylbenzene	ND		300	44	ug/L			09/08/18 12:22	100
N-Propylbenzene	ND		300	50	ug/L			09/08/18 12:22	100
o-Xylene	ND		200	15	ug/L			09/08/18 12:22	100
sec-Butylbenzene	ND		300	49	ug/L			09/08/18 12:22	100
Styrene	ND		500	51	ug/L			09/08/18 12:22	100
t-Butylbenzene	ND		300	58	ug/L			09/08/18 12:22	100
Tetrachloroethene	ND		300	41	ug/L			09/08/18 12:22	100
Toluene	ND		200	39	ug/L			09/08/18 12:22	100
trans-1,2-Dichloroethene	ND		300	39	ug/L			09/08/18 12:22	100
trans-1,3-Dichloropropene	ND		100	16	ug/L			09/08/18 12:22	100
Trichloroethene	ND		300	85	ug/L			09/08/18 12:22	100
Trichlorofluoromethane	ND		300	63	ug/L			09/08/18 12:22	100
Vinyl chloride	ND		100	22	ug/L			09/08/18 12:22	100

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*87*		*80 - 126*		*09/08/18 12:22*	*100*
1,2-Dichloroethane-d4 (Surr)	*105*		*80 - 126*		*09/18/18 16:28*	*100*
4-Bromofluorobenzene (Surr)	*94*		*80 - 125*		*09/08/18 12:22*	*100*
4-Bromofluorobenzene (Surr)	*104*		*80 - 125*		*09/18/18 16:28*	*100*
Dibromofluoromethane (Surr)	*96*		*77 - 120*		*09/08/18 12:22*	*100*
Dibromofluoromethane (Surr)	*99*		*77 - 120*		*09/18/18 16:28*	*100*
Toluene-d8 (Surr)	*104*		*80 - 122*		*09/08/18 12:22*	*100*
Toluene-d8 (Surr)	*105*		*80 - 122*		*09/18/18 16:28*	*100*
Trifluorotoluene (Surr)	*106*		*80 - 120*		*09/08/18 12:22*	*100*
Trifluorotoluene (Surr)	*104*		*80 - 120*		*09/18/18 16:28*	*100*

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Phenol	ND	*	20	1.8	ug/L		09/13/18 10:05	09/13/18 22:57	1
Bis(2-chloroethyl)ether	ND	*	3.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Chlorophenol	ND	*	5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
1,3-Dichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
1,4-Dichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzyl alcohol	ND		15	0.75	ug/L		09/13/18 10:05	09/13/18 22:57	1
1,2-Dichlorobenzene	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Methylphenol	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
3 & 4 Methylphenol	ND		4.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:57	1
N-Nitrosodi-n-propylamine	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
Hexachloroethane	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
Nitrobenzene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Isophorone	ND	*	2.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Nitrophenol	ND		5.0	0.75	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4-Dimethylphenol	ND		20	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzoic acid	ND		20	3.5	ug/L		09/13/18 10:05	09/13/18 22:57	1
Bis(2-chloroethoxy)methane	ND	*	3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4-Dichlorophenol	ND	*	20	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
1,2,4-Trichlorobenzene	ND	*	2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-3
Date Collected: 08/23/18 14:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-4
Matrix: Solid

Method: 8270D - Semivolatile Organic Compounds (GC/MS) - SPLP West (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Naphthalene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Chloroaniline	ND		50	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
Hexachlorobutadiene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Chloro-3-methylphenol	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Methylnaphthalene	ND		2.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:57	1
Hexachlorocyclopentadiene	ND		25	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4,6-Trichlorophenol	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4,5-Trichlorophenol	ND		2.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Chloronaphthalene	ND		5.0	0.15	ug/L		09/13/18 10:05	09/13/18 22:57	1
2-Nitroaniline	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
Dimethyl phthalate	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
Acenaphthylene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,6-Dinitrotoluene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
3-Nitroaniline	ND		15	0.15	ug/L		09/13/18 10:05	09/13/18 22:57	1
Acenaphthene	ND	*	2.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4-Dinitrophenol	ND		25	1.4	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Nitrophenol	ND		75	2.6	ug/L		09/13/18 10:05	09/13/18 22:57	1
Dibenzofuran	ND		2.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
2,4-Dinitrotoluene	ND		5.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
Diethyl phthalate	ND		60	2.8	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Chlorophenyl phenyl ether	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
Fluorene	ND		10	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Nitroaniline	ND		10	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
4,6-Dinitro-2-methylphenol	ND		25	1.3	ug/L		09/13/18 10:05	09/13/18 22:57	1
N-Nitrosodiphenylamine	ND		75	16	ug/L		09/13/18 10:05	09/13/18 22:57	1
4-Bromophenyl phenyl ether	ND		3.0	0.10	ug/L		09/13/18 10:05	09/13/18 22:57	1
Hexachlorobenzene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Pentachlorophenol	ND		50	1.3	ug/L		09/13/18 10:05	09/13/18 22:57	1
Phenanthrene	ND		5.0	1.2	ug/L		09/13/18 10:05	09/13/18 22:57	1
Anthracene	ND		75	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
Di-n-butyl phthalate	ND		15	2.2	ug/L		09/13/18 10:05	09/13/18 22:57	1
Fluoranthene	ND	*	15	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
Pyrene	ND	*	10	0.55	ug/L		09/13/18 10:05	09/13/18 22:57	1
Butyl benzyl phthalate	ND	*	50	4.7	ug/L		09/13/18 10:05	09/13/18 22:57	1
3,3'-Dichlorobenzidine	ND		75	0.95	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzo[a]anthracene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
Chrysene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Bis(2-ethylhexyl) phthalate	ND		75	13	ug/L		09/13/18 10:05	09/13/18 22:57	1
Di-n-octyl phthalate	ND		5.0	0.65	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzo[a]pyrene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Indeno[1,2,3-cd]pyrene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1
Dibenz(a,h)anthracene	ND	*	3.0	0.35	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzo[g,h,i]perylene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Carbazole	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 22:57	1
1-Methylnaphthalene	ND		5.0	0.10	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzo[b]fluoranthene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 22:57	1
Benzo[k]fluoranthene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 22:57	1
bis(chloroisopropyl) ether	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 22:57	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-3
Date Collected: 08/23/18 14:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-4
Matrix: Solid

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
2-Fluorophenol (Surr)	55		20 - 147	09/13/18 10:05	09/13/18 22:57	1
Phenol-d5 (Surr)	42		21 - 135	09/13/18 10:05	09/13/18 22:57	1
Nitrobenzene-d5 (Surr)	92		60 - 120	09/13/18 10:05	09/13/18 22:57	1
2-Fluorobiphenyl	58	X	63 - 120	09/13/18 10:05	09/13/18 22:57	1
2,4,6-Tribromophenol (Surr)	89		28 - 131	09/13/18 10:05	09/13/18 22:57	1
Terphenyl-d14 (Surr)	94		66 - 120	09/13/18 10:05	09/13/18 22:57	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-1
Date Collected: 08/23/18 14:50
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-5
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 09:28	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 09:28	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 09:28	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 09:28	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 09:28	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 09:28	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 09:28	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 09:28	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 09:28	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 09:28	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 09:28	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 09:28	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 09:28	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 09:28	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 09:28	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 09:28	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 09:28	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 09:28	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 09:28	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 09:28	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 09:28	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 09:28	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 09:28	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 09:28	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 09:28	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 09:28	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 09:28	1
Acetone	ND		50	7.8	ug/L			09/01/18 09:28	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 09:28	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 09:28	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 09:28	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 09:28	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 09:28	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 09:28	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 09:28	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 09:28	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 09:28	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 09:28	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 09:28	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 09:28	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 09:28	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 09:28	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 09:28	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 09:28	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 09:28	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 09:28	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 09:28	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 09:28	1
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 09:28	1

Client Sample ID: SITK-18-PW-1
Date Collected: 08/23/18 14:50
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-5
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 09:28	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 09:28	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 09:28	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 09:28	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 09:28	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 09:28	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 09:28	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 09:28	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 09:28	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 09:28	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 09:28	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 09:28	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 09:28	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 09:28	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 09:28	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 09:28	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*99*		*80 - 126*		*09/01/18 09:28*	*1*
4-Bromofluorobenzene (Surr)	*98*		*80 - 125*		*09/01/18 09:28*	*1*
Dibromofluoromethane (Surr)	*102*		*77 - 120*		*09/01/18 09:28*	*1*
Toluene-d8 (Surr)	*100*		*80 - 122*		*09/01/18 09:28*	*1*
Trifluorotoluene (Surr)	*104*		*80 - 120*		*09/01/18 09:28*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,2,4-Trichlorobenzene	ND		0.41	0.041	ug/L		08/29/18 12:59	09/04/18 20:24	1
1,2-Dichlorobenzene	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
1,3-Dichlorobenzene	ND		0.41	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
1,4-Dichlorobenzene	ND		0.41	0.061	ug/L		08/29/18 12:59	09/04/18 20:24	1
1-Methylnaphthalene	ND		1.0	0.071	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4,5-Trichlorophenol	ND		0.41	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4,6-Trichlorophenol	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4-Dichlorophenol	ND		4.1	0.54	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4-Dimethylphenol	ND		4.1	0.84	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4-Dinitrophenol	ND		5.1	1.0	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,4-Dinitrotoluene	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 20:24	1
2,6-Dinitrotoluene	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Chloronaphthalene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Chlorophenol	ND		0.61	0.22	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Methylnaphthalene	ND		0.41	0.061	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Methylphenol	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Nitroaniline	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
2-Nitrophenol	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 20:24	1
3 & 4 Methylphenol	ND		0.81	0.18	ug/L		08/29/18 12:59	09/04/18 20:24	1
3,3'-Dichlorobenzidine	ND		15	1.1	ug/L		08/29/18 12:59	09/04/18 20:24	1
3-Nitroaniline	ND		3.0	0.74	ug/L		08/29/18 12:59	09/04/18 20:24	1
4,6-Dinitro-2-methylphenol	ND		5.1	1.0	ug/L		08/29/18 12:59	09/04/18 20:24	1
4-Bromophenyl phenyl ether	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
4-Chloro-3-methylphenol	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1

Client Sample ID: SITK-18-PW-1
Date Collected: 08/23/18 14:50
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-5
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4-Chloroaniline	ND	*	10	2.1	ug/L		08/29/18 12:59	09/04/18 20:24	1
4-Chlorophenyl phenyl ether	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
4-Nitroaniline	ND		2.0	0.29	ug/L		08/29/18 12:59	09/04/18 20:24	1
4-Nitrophenol	ND		15	1.8	ug/L		08/29/18 12:59	09/04/18 20:24	1
Acenaphthene	ND		0.41	0.081	ug/L		08/29/18 12:59	09/04/18 20:24	1
Acenaphthylene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Anthracene	ND		15	0.020	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzo[a]anthracene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzo[a]pyrene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzo[b]fluoranthene	ND		1.0	0.051	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzo[g,h,i]perylene	ND		1.0	0.051	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzo[k]fluoranthene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzoic acid	ND		4.1	0.86	ug/L		08/29/18 12:59	09/04/18 20:24	1
Benzyl alcohol	ND	*	3.0	0.70	ug/L		08/29/18 12:59	09/04/18 20:24	1
Bis(2-chloroethoxy)methane	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Bis(2-chloroethyl)ether	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Bis(2-ethylhexyl) phthalate	ND	*	15	6.4	ug/L		08/29/18 12:59	09/04/18 20:24	1
bis(chloroisopropyl) ether	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Butyl benzyl phthalate	ND		10	4.0	ug/L		08/29/18 12:59	09/04/18 20:24	1
Carbazole	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Chrysene	ND		0.61	0.17	ug/L		08/29/18 12:59	09/04/18 20:24	1
Dibenz(a,h)anthracene	ND		0.61	0.020	ug/L		08/29/18 12:59	09/04/18 20:24	1
Dibenzofuran	ND		0.41	0.061	ug/L		08/29/18 12:59	09/04/18 20:24	1
Diethyl phthalate	ND		12	2.9	ug/L		08/29/18 12:59	09/04/18 20:24	1
Dimethyl phthalate	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Di-n-butyl phthalate	ND		3.0	0.56	ug/L		08/29/18 12:59	09/04/18 20:24	1
Di-n-octyl phthalate	ND		1.0	0.18	ug/L		08/29/18 12:59	09/04/18 20:24	1
Fluoranthene	ND		3.0	0.15	ug/L		08/29/18 12:59	09/04/18 20:24	1
Fluorene	ND		2.0	0.091	ug/L		08/29/18 12:59	09/04/18 20:24	1
Hexachlorobenzene	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Hexachlorobutadiene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Hexachlorocyclopentadiene	ND	*	3.0	1.3	ug/L		08/29/18 12:59	09/04/18 20:24	1
Hexachloroethane	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Indeno[1,2,3-cd]pyrene	ND		1.0	0.051	ug/L		08/29/18 12:59	09/04/18 20:24	1
Isophorone	ND		0.41	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Naphthalene	ND		0.41	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
Nitrobenzene	**0.76**		0.61	0.22	ug/L		08/29/18 12:59	09/04/18 20:24	1
N-Nitrosodi-n-propylamine	ND		0.61	0.10	ug/L		08/29/18 12:59	09/04/18 20:24	1
N-Nitrosodiphenylamine	ND		3.0	0.61	ug/L		08/29/18 12:59	09/04/18 20:24	1
Pentachlorophenol	ND		10	2.5	ug/L		08/29/18 12:59	09/04/18 20:24	1
Phenanthrene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 20:24	1
Phenol	ND		4.1	0.93	ug/L		08/29/18 12:59	09/04/18 20:24	1
Pyrene	ND		2.0	0.40	ug/L		08/29/18 12:59	09/04/18 20:24	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*83*		*48 - 125*	*08/29/18 12:59*	*09/04/18 20:24*	*1*
2-Fluorobiphenyl	*92*		*50 - 120*	*08/29/18 12:59*	*09/04/18 20:24*	*1*
2-Fluorophenol (Surr)	*81*		*50 - 120*	*08/29/18 12:59*	*09/04/18 20:24*	*1*
Nitrobenzene-d5 (Surr)	*100*		*62 - 120*	*08/29/18 12:59*	*09/04/18 20:24*	*1*
Phenol-d5 (Surr)	*82*		*52 - 120*	*08/29/18 12:59*	*09/04/18 20:24*	*1*

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-1
Date Collected: 08/23/18 14:50
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-5
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
Terphenyl-d14 (Surr)	101		55 - 126	08/29/18 12:59	09/04/18 20:24	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-4
Date Collected: 08/23/18 15:15
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-6
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 09:54	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 09:54	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 09:54	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 09:54	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 09:54	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 09:54	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 09:54	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 09:54	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 09:54	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 09:54	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 09:54	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 09:54	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 09:54	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 09:54	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 09:54	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 09:54	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 09:54	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 09:54	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 09:54	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 09:54	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 09:54	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 09:54	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 09:54	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 09:54	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 09:54	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 09:54	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 09:54	1
Acetone	ND		50	7.8	ug/L			09/01/18 09:54	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 09:54	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 09:54	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 09:54	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 09:54	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 09:54	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 09:54	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 09:54	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 09:54	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 09:54	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 09:54	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 09:54	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 09:54	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 09:54	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 09:54	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 09:54	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 09:54	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 09:54	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 09:54	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 09:54	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 09:54	1
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 09:54	1

Client Sample ID: SITK-18-PW-4
Date Collected: 08/23/18 15:15
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-6
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 09:54	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 09:54	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 09:54	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 09:54	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 09:54	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 09:54	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 09:54	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 09:54	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 09:54	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 09:54	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 09:54	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 09:54	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 09:54	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 09:54	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 09:54	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 09:54	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*100*		*80 - 126*		*09/01/18 09:54*	*1*
4-Bromofluorobenzene (Surr)	*100*		*80 - 125*		*09/01/18 09:54*	*1*
Dibromofluoromethane (Surr)	*103*		*77 - 120*		*09/01/18 09:54*	*1*
Toluene-d8 (Surr)	*102*		*80 - 122*		*09/01/18 09:54*	*1*
Trifluorotoluene (Surr)	*103*		*80 - 120*		*09/01/18 09:54*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,2,4-Trichlorobenzene	ND		0.40	0.040	ug/L		08/29/18 12:59	09/04/18 20:48	1
1,2-Dichlorobenzene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
1,3-Dichlorobenzene	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
1,4-Dichlorobenzene	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 20:48	1
1-Methylnaphthalene	ND		1.0	0.070	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4,5-Trichlorophenol	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4,6-Trichlorophenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4-Dichlorophenol	ND		4.0	0.53	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4-Dimethylphenol	ND		4.0	0.83	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4-Dinitrophenol	ND		5.0	1.0	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,4-Dinitrotoluene	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 20:48	1
2,6-Dinitrotoluene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Chloronaphthalene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Chlorophenol	ND		0.60	0.22	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Methylnaphthalene	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Methylphenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Nitroaniline	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
2-Nitrophenol	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 20:48	1
3 & 4 Methylphenol	ND		0.80	0.18	ug/L		08/29/18 12:59	09/04/18 20:48	1
3,3'-Dichlorobenzidine	ND		15	1.1	ug/L		08/29/18 12:59	09/04/18 20:48	1
3-Nitroaniline	ND		3.0	0.73	ug/L		08/29/18 12:59	09/04/18 20:48	1
4,6-Dinitro-2-methylphenol	ND		5.0	1.0	ug/L		08/29/18 12:59	09/04/18 20:48	1
4-Bromophenyl phenyl ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
4-Chloro-3-methylphenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1

Client Sample ID: SITK-18-PW-4
Date Collected: 08/23/18 15:15
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-6
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4-Chloroaniline	ND	*	10	2.1	ug/L		08/29/18 12:59	09/04/18 20:48	1
4-Chlorophenyl phenyl ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
4-Nitroaniline	ND		2.0	0.29	ug/L		08/29/18 12:59	09/04/18 20:48	1
4-Nitrophenol	ND		15	1.8	ug/L		08/29/18 12:59	09/04/18 20:48	1
Acenaphthene	ND		0.40	0.080	ug/L		08/29/18 12:59	09/04/18 20:48	1
Acenaphthylene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Anthracene	ND		15	0.020	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzo[a]anthracene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzo[a]pyrene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzo[b]fluoranthene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzo[g,h,i]perylene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzo[k]fluoranthene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzoic acid	ND		4.0	0.85	ug/L		08/29/18 12:59	09/04/18 20:48	1
Benzyl alcohol	ND	*	3.0	0.69	ug/L		08/29/18 12:59	09/04/18 20:48	1
Bis(2-chloroethoxy)methane	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Bis(2-chloroethyl)ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Bis(2-ethylhexyl) phthalate	ND	*	15	6.3	ug/L		08/29/18 12:59	09/04/18 20:48	1
bis(chloroisopropyl) ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Butyl benzyl phthalate	ND		10	4.0	ug/L		08/29/18 12:59	09/04/18 20:48	1
Carbazole	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Chrysene	ND		0.60	0.17	ug/L		08/29/18 12:59	09/04/18 20:48	1
Dibenz(a,h)anthracene	ND		0.60	0.020	ug/L		08/29/18 12:59	09/04/18 20:48	1
Dibenzofuran	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 20:48	1
Diethyl phthalate	ND		12	2.8	ug/L		08/29/18 12:59	09/04/18 20:48	1
Dimethyl phthalate	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Di-n-butyl phthalate	ND		3.0	0.55	ug/L		08/29/18 12:59	09/04/18 20:48	1
Di-n-octyl phthalate	ND		1.0	0.18	ug/L		08/29/18 12:59	09/04/18 20:48	1
Fluoranthene	ND		3.0	0.15	ug/L		08/29/18 12:59	09/04/18 20:48	1
Fluorene	ND		2.0	0.090	ug/L		08/29/18 12:59	09/04/18 20:48	1
Hexachlorobenzene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Hexachlorobutadiene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Hexachlorocyclopentadiene	ND	*	3.0	1.3	ug/L		08/29/18 12:59	09/04/18 20:48	1
Hexachloroethane	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Indeno[1,2,3-cd]pyrene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 20:48	1
Isophorone	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Naphthalene	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
Nitrobenzene	ND		0.60	0.22	ug/L		08/29/18 12:59	09/04/18 20:48	1
N-Nitrosodi-n-propylamine	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 20:48	1
N-Nitrosodiphenylamine	ND		3.0	0.60	ug/L		08/29/18 12:59	09/04/18 20:48	1
Pentachlorophenol	ND		10	2.5	ug/L		08/29/18 12:59	09/04/18 20:48	1
Phenanthrene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 20:48	1
Phenol	ND		4.0	0.92	ug/L		08/29/18 12:59	09/04/18 20:48	1
Pyrene	ND		2.0	0.39	ug/L		08/29/18 12:59	09/04/18 20:48	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*82*		*48 - 125*	*08/29/18 12:59*	*09/04/18 20:48*	*1*
2-Fluorobiphenyl	*98*		*50 - 120*	*08/29/18 12:59*	*09/04/18 20:48*	*1*
2-Fluorophenol (Surr)	*81*		*50 - 120*	*08/29/18 12:59*	*09/04/18 20:48*	*1*
Nitrobenzene-d5 (Surr)	*101*		*62 - 120*	*08/29/18 12:59*	*09/04/18 20:48*	*1*
Phenol-d5 (Surr)	*83*		*52 - 120*	*08/29/18 12:59*	*09/04/18 20:48*	*1*

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-4
Date Collected: 08/23/18 15:15
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-6
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
Terphenyl-d14 (Surr)	103		55 - 126	08/29/18 12:59	09/04/18 20:48	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-2
Date Collected: 08/23/18 16:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-7
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 10:19	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 10:19	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 10:19	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 10:19	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 10:19	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 10:19	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 10:19	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 10:19	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 10:19	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 10:19	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 10:19	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 10:19	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 10:19	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 10:19	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 10:19	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 10:19	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 10:19	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 10:19	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 10:19	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 10:19	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 10:19	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 10:19	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 10:19	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 10:19	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 10:19	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 10:19	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 10:19	1
Acetone	ND		50	7.8	ug/L			09/01/18 10:19	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 10:19	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 10:19	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 10:19	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 10:19	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 10:19	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 10:19	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 10:19	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 10:19	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 10:19	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 10:19	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 10:19	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 10:19	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 10:19	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 10:19	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 10:19	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 10:19	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 10:19	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 10:19	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 10:19	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 10:19	1
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 10:19	1

Client Sample ID: SITK-18-PW-2
Date Collected: 08/23/18 16:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-7
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 10:19	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 10:19	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 10:19	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 10:19	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 10:19	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 10:19	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 10:19	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 10:19	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 10:19	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 10:19	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 10:19	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 10:19	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 10:19	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 10:19	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 10:19	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 10:19	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*100*		*80 - 126*		*09/01/18 10:19*	*1*
4-Bromofluorobenzene (Surr)	*97*		*80 - 125*		*09/01/18 10:19*	*1*
Dibromofluoromethane (Surr)	*100*		*77 - 120*		*09/01/18 10:19*	*1*
Toluene-d8 (Surr)	*102*		*80 - 122*		*09/01/18 10:19*	*1*
Trifluorotoluene (Surr)	*102*		*80 - 120*		*09/01/18 10:19*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,2,4-Trichlorobenzene	ND		0.39	0.039	ug/L		08/29/18 12:59	09/04/18 21:13	1
1,2-Dichlorobenzene	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
1,3-Dichlorobenzene	ND		0.39	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
1,4-Dichlorobenzene	ND		0.39	0.059	ug/L		08/29/18 12:59	09/04/18 21:13	1
1-Methylnaphthalene	ND		0.98	0.069	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4,5-Trichlorophenol	ND		0.39	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4,6-Trichlorophenol	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4-Dichlorophenol	ND		3.9	0.52	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4-Dimethylphenol	ND		3.9	0.82	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4-Dinitrophenol	ND		4.9	0.98	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,4-Dinitrotoluene	ND		0.98	0.14	ug/L		08/29/18 12:59	09/04/18 21:13	1
2,6-Dinitrotoluene	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Chloronaphthalene	ND		0.98	0.13	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Chlorophenol	ND		0.59	0.22	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Methylnaphthalene	ND		0.39	0.059	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Methylphenol	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Nitroaniline	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
2-Nitrophenol	ND		0.98	0.14	ug/L		08/29/18 12:59	09/04/18 21:13	1
3 & 4 Methylphenol	ND		0.79	0.18	ug/L		08/29/18 12:59	09/04/18 21:13	1
3,3'-Dichlorobenzidine	ND		15	1.0	ug/L		08/29/18 12:59	09/04/18 21:13	1
3-Nitroaniline	ND		3.0	0.72	ug/L		08/29/18 12:59	09/04/18 21:13	1
4,6-Dinitro-2-methylphenol	ND		4.9	0.98	ug/L		08/29/18 12:59	09/04/18 21:13	1
4-Bromophenyl phenyl ether	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
4-Chloro-3-methylphenol	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1

Client Sample ID: SITK-18-PW-2
Date Collected: 08/23/18 16:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-7
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4-Chloroaniline	ND	*	9.8	2.1	ug/L		08/29/18 12:59	09/04/18 21:13	1
4-Chlorophenyl phenyl ether	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
4-Nitroaniline	ND		2.0	0.29	ug/L		08/29/18 12:59	09/04/18 21:13	1
4-Nitrophenol	ND		15	1.8	ug/L		08/29/18 12:59	09/04/18 21:13	1
Acenaphthene	ND		0.39	0.079	ug/L		08/29/18 12:59	09/04/18 21:13	1
Acenaphthylene	ND		0.98	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Anthracene	ND		15	0.020	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzo[a]anthracene	ND		0.98	0.020	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzo[a]pyrene	ND		0.98	0.020	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzo[b]fluoranthene	ND		0.98	0.049	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzo[g,h,i]perylene	ND		0.98	0.049	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzo[k]fluoranthene	ND		0.98	0.020	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzoic acid	ND		3.9	0.84	ug/L		08/29/18 12:59	09/04/18 21:13	1
Benzyl alcohol	ND	*	3.0	0.68	ug/L		08/29/18 12:59	09/04/18 21:13	1
Bis(2-chloroethoxy)methane	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Bis(2-chloroethyl)ether	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Bis(2-ethylhexyl) phthalate	ND	*	15	6.2	ug/L		08/29/18 12:59	09/04/18 21:13	1
bis(chloroisopropyl) ether	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Butyl benzyl phthalate	ND		9.8	3.9	ug/L		08/29/18 12:59	09/04/18 21:13	1
Carbazole	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Chrysene	ND		0.59	0.17	ug/L		08/29/18 12:59	09/04/18 21:13	1
Dibenz(a,h)anthracene	ND		0.59	0.020	ug/L		08/29/18 12:59	09/04/18 21:13	1
Dibenzofuran	ND		0.39	0.059	ug/L		08/29/18 12:59	09/04/18 21:13	1
Diethyl phthalate	ND		12	2.8	ug/L		08/29/18 12:59	09/04/18 21:13	1
Dimethyl phthalate	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Di-n-butyl phthalate	ND		3.0	0.54	ug/L		08/29/18 12:59	09/04/18 21:13	1
Di-n-octyl phthalate	ND		0.98	0.18	ug/L		08/29/18 12:59	09/04/18 21:13	1
Fluoranthene	ND		3.0	0.15	ug/L		08/29/18 12:59	09/04/18 21:13	1
Fluorene	ND		2.0	0.089	ug/L		08/29/18 12:59	09/04/18 21:13	1
Hexachlorobenzene	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Hexachlorobutadiene	ND		0.98	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Hexachlorocyclopentadiene	ND	*	3.0	1.3	ug/L		08/29/18 12:59	09/04/18 21:13	1
Hexachloroethane	ND		0.98	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Indeno[1,2,3-cd]pyrene	ND		0.98	0.049	ug/L		08/29/18 12:59	09/04/18 21:13	1
Isophorone	ND		0.39	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Naphthalene	ND		0.39	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
Nitrobenzene	ND		0.59	0.22	ug/L		08/29/18 12:59	09/04/18 21:13	1
N-Nitrosodi-n-propylamine	ND		0.59	0.098	ug/L		08/29/18 12:59	09/04/18 21:13	1
N-Nitrosodiphenylamine	ND		3.0	0.59	ug/L		08/29/18 12:59	09/04/18 21:13	1
Pentachlorophenol	ND		9.8	2.4	ug/L		08/29/18 12:59	09/04/18 21:13	1
Phenanthrene	ND		0.98	0.13	ug/L		08/29/18 12:59	09/04/18 21:13	1
Phenol	ND		3.9	0.91	ug/L		08/29/18 12:59	09/04/18 21:13	1
Pyrene	ND		2.0	0.38	ug/L		08/29/18 12:59	09/04/18 21:13	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*88*		*48 - 125*	*08/29/18 12:59*	*09/04/18 21:13*	*1*
2-Fluorobiphenyl	*94*		*50 - 120*	*08/29/18 12:59*	*09/04/18 21:13*	*1*
2-Fluorophenol (Surr)	*76*		*50 - 120*	*08/29/18 12:59*	*09/04/18 21:13*	*1*
Nitrobenzene-d5 (Surr)	*97*		*62 - 120*	*08/29/18 12:59*	*09/04/18 21:13*	*1*
Phenol-d5 (Surr)	*79*		*52 - 120*	*08/29/18 12:59*	*09/04/18 21:13*	*1*

Client Sample ID: SITK-18-PW-2
Date Collected: 08/23/18 16:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-7
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
Terphenyl-d14 (Surr)	102		55 - 126	08/29/18 12:59	09/04/18 21:13	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-3
Date Collected: 08/23/18 16:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-8
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 10:45	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 10:45	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 10:45	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 10:45	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 10:45	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 10:45	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 10:45	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 10:45	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 10:45	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 10:45	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 10:45	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 10:45	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 10:45	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 10:45	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 10:45	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 10:45	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 10:45	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 10:45	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 10:45	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 10:45	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 10:45	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 10:45	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 10:45	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 10:45	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 10:45	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 10:45	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 10:45	1
Acetone	ND		50	7.8	ug/L			09/01/18 10:45	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 10:45	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 10:45	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 10:45	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 10:45	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 10:45	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 10:45	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 10:45	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 10:45	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 10:45	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 10:45	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 10:45	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 10:45	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 10:45	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 10:45	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 10:45	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 10:45	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 10:45	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 10:45	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 10:45	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 10:45	1
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 10:45	1

Client Sample ID: SITK-18-PW-3
Date Collected: 08/23/18 16:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-8
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 10:45	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 10:45	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 10:45	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 10:45	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 10:45	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 10:45	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 10:45	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 10:45	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 10:45	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 10:45	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 10:45	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 10:45	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 10:45	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 10:45	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 10:45	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 10:45	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*101*		*80 - 126*		*09/01/18 10:45*	*1*
4-Bromofluorobenzene (Surr)	*98*		*80 - 125*		*09/01/18 10:45*	*1*
Dibromofluoromethane (Surr)	*101*		*77 - 120*		*09/01/18 10:45*	*1*
Toluene-d8 (Surr)	*101*		*80 - 122*		*09/01/18 10:45*	*1*
Trifluorotoluene (Surr)	*102*		*80 - 120*		*09/01/18 10:45*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,2,4-Trichlorobenzene	ND		0.39	0.039	ug/L		08/29/18 12:59	09/04/18 21:38	1
1,2-Dichlorobenzene	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
1,3-Dichlorobenzene	ND		0.39	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
1,4-Dichlorobenzene	ND		0.39	0.058	ug/L		08/29/18 12:59	09/04/18 21:38	1
1-Methylnaphthalene	ND		0.97	0.068	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4,5-Trichlorophenol	ND		0.39	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4,6-Trichlorophenol	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4-Dichlorophenol	ND		3.9	0.51	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4-Dimethylphenol	ND		3.9	0.80	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4-Dinitrophenol	ND		4.8	0.97	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,4-Dinitrotoluene	ND		0.97	0.14	ug/L		08/29/18 12:59	09/04/18 21:38	1
2,6-Dinitrotoluene	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Chloronaphthalene	ND		0.97	0.13	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Chlorophenol	ND		0.58	0.21	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Methylnaphthalene	ND		0.39	0.058	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Methylphenol	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Nitroaniline	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
2-Nitrophenol	ND		0.97	0.14	ug/L		08/29/18 12:59	09/04/18 21:38	1
3 & 4 Methylphenol	ND		0.77	0.17	ug/L		08/29/18 12:59	09/04/18 21:38	1
3,3'-Dichlorobenzidine	ND		15	1.0	ug/L		08/29/18 12:59	09/04/18 21:38	1
3-Nitroaniline	ND		2.9	0.71	ug/L		08/29/18 12:59	09/04/18 21:38	1
4,6-Dinitro-2-methylphenol	ND		4.8	0.97	ug/L		08/29/18 12:59	09/04/18 21:38	1
4-Bromophenyl phenyl ether	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
4-Chloro-3-methylphenol	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1

Client Sample ID: SITK-18-PW-3
Date Collected: 08/23/18 16:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-8
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4-Chloroaniline	ND	*	9.7	2.0	ug/L		08/29/18 12:59	09/04/18 21:38	1
4-Chlorophenyl phenyl ether	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
4-Nitroaniline	ND		1.9	0.28	ug/L		08/29/18 12:59	09/04/18 21:38	1
4-Nitrophenol	ND		15	1.7	ug/L		08/29/18 12:59	09/04/18 21:38	1
Acenaphthene	ND		0.39	0.077	ug/L		08/29/18 12:59	09/04/18 21:38	1
Acenaphthylene	ND		0.97	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Anthracene	ND		15	0.019	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzo[a]anthracene	ND		0.97	0.019	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzo[a]pyrene	ND		0.97	0.019	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzo[b]fluoranthene	ND		0.97	0.048	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzo[g,h,i]perylene	ND		0.97	0.048	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzo[k]fluoranthene	ND		0.97	0.019	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzoic acid	ND		3.9	0.82	ug/L		08/29/18 12:59	09/04/18 21:38	1
Benzyl alcohol	ND	*	2.9	0.67	ug/L		08/29/18 12:59	09/04/18 21:38	1
Bis(2-chloroethoxy)methane	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Bis(2-chloroethyl)ether	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Bis(2-ethylhexyl) phthalate	ND	*	15	6.1	ug/L		08/29/18 12:59	09/04/18 21:38	1
bis(chloroisopropyl) ether	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Butyl benzyl phthalate	ND		9.7	3.9	ug/L		08/29/18 12:59	09/04/18 21:38	1
Carbazole	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Chrysene	ND		0.58	0.16	ug/L		08/29/18 12:59	09/04/18 21:38	1
Dibenz(a,h)anthracene	ND		0.58	0.019	ug/L		08/29/18 12:59	09/04/18 21:38	1
Dibenzofuran	ND		0.39	0.058	ug/L		08/29/18 12:59	09/04/18 21:38	1
Diethyl phthalate	ND		12	2.7	ug/L		08/29/18 12:59	09/04/18 21:38	1
Dimethyl phthalate	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Di-n-butyl phthalate	ND		2.9	0.53	ug/L		08/29/18 12:59	09/04/18 21:38	1
Di-n-octyl phthalate	ND		0.97	0.17	ug/L		08/29/18 12:59	09/04/18 21:38	1
Fluoranthene	ND		2.9	0.15	ug/L		08/29/18 12:59	09/04/18 21:38	1
Fluorene	ND		1.9	0.087	ug/L		08/29/18 12:59	09/04/18 21:38	1
Hexachlorobenzene	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Hexachlorobutadiene	ND		0.97	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Hexachlorocyclopentadiene	ND	*	2.9	1.2	ug/L		08/29/18 12:59	09/04/18 21:38	1
Hexachloroethane	ND		0.97	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Indeno[1,2,3-cd]pyrene	ND		0.97	0.048	ug/L		08/29/18 12:59	09/04/18 21:38	1
Isophorone	ND		0.39	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Naphthalene	ND		0.39	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
Nitrobenzene	ND		0.58	0.21	ug/L		08/29/18 12:59	09/04/18 21:38	1
N-Nitrosodi-n-propylamine	ND		0.58	0.097	ug/L		08/29/18 12:59	09/04/18 21:38	1
N-Nitrosodiphenylamine	ND		2.9	0.58	ug/L		08/29/18 12:59	09/04/18 21:38	1
Pentachlorophenol	ND		9.7	2.4	ug/L		08/29/18 12:59	09/04/18 21:38	1
Phenanthrene	ND		0.97	0.13	ug/L		08/29/18 12:59	09/04/18 21:38	1
Phenol	ND		3.9	0.89	ug/L		08/29/18 12:59	09/04/18 21:38	1
Pyrene	ND		1.9	0.38	ug/L		08/29/18 12:59	09/04/18 21:38	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*84*		*48 - 125*	*08/29/18 12:59*	*09/04/18 21:38*	*1*
2-Fluorobiphenyl	*94*		*50 - 120*	*08/29/18 12:59*	*09/04/18 21:38*	*1*
2-Fluorophenol (Surr)	*79*		*50 - 120*	*08/29/18 12:59*	*09/04/18 21:38*	*1*
Nitrobenzene-d5 (Surr)	*98*		*62 - 120*	*08/29/18 12:59*	*09/04/18 21:38*	*1*
Phenol-d5 (Surr)	*81*		*52 - 120*	*08/29/18 12:59*	*09/04/18 21:38*	*1*

Client Sample ID: SITK-18-PW-3
Date Collected: 08/23/18 16:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-8
Matrix: Water

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Surrogate	%Recovery	Qualifier	Limits	Prepared	Analyzed	Dil Fac
Terphenyl-d14 (Surr)	101		55 - 126	08/29/18 12:59	09/04/18 21:38	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-TB-PW-1
Date Collected: 08/23/18 08:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-9
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 05:07	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 05:07	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 05:07	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 05:07	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 05:07	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 05:07	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 05:07	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 05:07	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 05:07	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 05:07	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 05:07	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 05:07	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 05:07	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 05:07	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 05:07	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 05:07	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 05:07	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 05:07	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 05:07	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 05:07	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 05:07	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 05:07	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 05:07	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 05:07	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 05:07	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 05:07	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 05:07	1
Acetone	ND		50	7.8	ug/L			09/01/18 05:07	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 05:07	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 05:07	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 05:07	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 05:07	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 05:07	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 05:07	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 05:07	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 05:07	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 05:07	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 05:07	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 05:07	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 05:07	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 05:07	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 05:07	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 05:07	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 05:07	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 05:07	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 05:07	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 05:07	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 05:07	1
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 05:07	1

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-TB-PW-1
Date Collected: 08/23/18 08:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-9
Matrix: Water

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 05:07	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 05:07	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 05:07	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 05:07	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 05:07	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 05:07	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 05:07	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 05:07	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 05:07	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 05:07	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 05:07	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 05:07	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 05:07	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 05:07	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 05:07	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 05:07	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*102*		*80 - 126*		*09/01/18 05:07*	*1*
4-Bromofluorobenzene (Surr)	*99*		*80 - 125*		*09/01/18 05:07*	*1*
Dibromofluoromethane (Surr)	*100*		*77 - 120*		*09/01/18 05:07*	*1*
Toluene-d8 (Surr)	*100*		*80 - 122*		*09/01/18 05:07*	*1*
Trifluorotoluene (Surr)	*104*		*80 - 120*		*09/01/18 05:07*	*1*

Method: 8260C - Volatile Organic Compounds by GC/MS

Lab Sample ID: MB 580-283011/5
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Method Blank
Prep Type: Total/NA

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		2.0	0.18	ug/L			09/01/18 03:50	1
1,1,1-Trichloroethane	ND		3.0	0.14	ug/L			09/01/18 03:50	1
1,1,2,2-Tetrachloroethane	ND		3.0	0.52	ug/L			09/01/18 03:50	1
1,1,2-Trichloroethane	ND		1.0	0.24	ug/L			09/01/18 03:50	1
1,1-Dichloroethane	ND		2.0	0.22	ug/L			09/01/18 03:50	1
1,1-Dichloroethene	ND		4.0	0.78	ug/L			09/01/18 03:50	1
1,1-Dichloropropene	ND		3.0	0.29	ug/L			09/01/18 03:50	1
1,2,3-Trichlorobenzene	ND		5.0	0.46	ug/L			09/01/18 03:50	1
1,2,3-Trichloropropane	ND		2.0	0.41	ug/L			09/01/18 03:50	1
1,2,4-Trichlorobenzene	ND		2.0	0.33	ug/L			09/01/18 03:50	1
1,2,4-Trimethylbenzene	ND		3.0	0.61	ug/L			09/01/18 03:50	1
1,2-Dibromo-3-Chloropropane	ND		10	1.8	ug/L			09/01/18 03:50	1
1,2-Dibromoethane	ND		2.0	0.40	ug/L			09/01/18 03:50	1
1,2-Dichlorobenzene	ND		2.0	0.46	ug/L			09/01/18 03:50	1
1,2-Dichloroethane	ND		2.0	0.53	ug/L			09/01/18 03:50	1
1,2-Dichloropropane	ND		1.0	0.18	ug/L			09/01/18 03:50	1
1,3,5-Trimethylbenzene	ND		3.0	0.55	ug/L			09/01/18 03:50	1
1,3-Dichlorobenzene	ND		2.0	0.18	ug/L			09/01/18 03:50	1
1,3-Dichloropropane	ND		2.0	0.35	ug/L			09/01/18 03:50	1
1,4-Dichlorobenzene	ND		4.0	0.98	ug/L			09/01/18 03:50	1
2,2-Dichloropropane	ND		3.0	0.32	ug/L			09/01/18 03:50	1
2-Butanone	ND		20	4.7	ug/L			09/01/18 03:50	1
2-Chlorotoluene	ND		3.0	0.51	ug/L			09/01/18 03:50	1
2-Hexanone	ND		20	4.0	ug/L			09/01/18 03:50	1
4-Chlorotoluene	ND		2.0	0.51	ug/L			09/01/18 03:50	1
4-Isopropyltoluene	ND		3.0	0.28	ug/L			09/01/18 03:50	1
4-Methyl-2-pentanone	ND		15	2.5	ug/L			09/01/18 03:50	1
Acetone	ND		50	7.8	ug/L			09/01/18 03:50	1
Benzene	ND		3.0	0.53	ug/L			09/01/18 03:50	1
Bromobenzene	ND		2.0	0.18	ug/L			09/01/18 03:50	1
Bromochloromethane	ND		2.0	0.29	ug/L			09/01/18 03:50	1
Bromodichloromethane	ND		2.0	0.14	ug/L			09/01/18 03:50	1
Bromoform	ND		3.0	0.56	ug/L			09/01/18 03:50	1
Bromomethane	ND		6.0	1.1	ug/L			09/01/18 03:50	1
Carbon disulfide	ND		3.0	0.53	ug/L			09/01/18 03:50	1
Carbon tetrachloride	ND		3.0	0.30	ug/L			09/01/18 03:50	1
Chlorobenzene	ND		2.0	0.44	ug/L			09/01/18 03:50	1
Chloroethane	ND		5.0	1.1	ug/L			09/01/18 03:50	1
Chloroform	ND		5.0	0.50	ug/L			09/01/18 03:50	1
Chloromethane	ND		20	5.4	ug/L			09/01/18 03:50	1
cis-1,2-Dichloroethene	ND		3.0	0.69	ug/L			09/01/18 03:50	1
cis-1,3-Dichloropropene	ND		1.0	0.20	ug/L			09/01/18 03:50	1
Dibromochloromethane	ND		1.0	0.20	ug/L			09/01/18 03:50	1
Dibromomethane	ND		2.0	0.34	ug/L			09/01/18 03:50	1
Dichlorodifluoromethane	ND		10	2.3	ug/L			09/01/18 03:50	1
Ethylbenzene	ND		3.0	0.50	ug/L			09/01/18 03:50	1
Hexachlorobutadiene	ND		6.0	0.79	ug/L			09/01/18 03:50	1
Isopropylbenzene	ND		2.0	0.51	ug/L			09/01/18 03:50	1

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: MB 580-283011/5
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Method Blank
Prep Type: Total/NA

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Methyl tert-butyl ether	ND		2.0	0.44	ug/L			09/01/18 03:50	1
Methylene Chloride	ND		5.0	1.4	ug/L			09/01/18 03:50	1
m-Xylene & p-Xylene	ND		3.0	0.75	ug/L			09/01/18 03:50	1
Naphthalene	ND		4.0	0.93	ug/L			09/01/18 03:50	1
n-Butylbenzene	ND		3.0	0.44	ug/L			09/01/18 03:50	1
N-Propylbenzene	ND		3.0	0.50	ug/L			09/01/18 03:50	1
o-Xylene	ND		2.0	0.15	ug/L			09/01/18 03:50	1
sec-Butylbenzene	ND		3.0	0.49	ug/L			09/01/18 03:50	1
Styrene	ND		5.0	0.51	ug/L			09/01/18 03:50	1
t-Butylbenzene	ND		3.0	0.58	ug/L			09/01/18 03:50	1
Tetrachloroethene	ND		3.0	0.41	ug/L			09/01/18 03:50	1
Toluene	ND		2.0	0.39	ug/L			09/01/18 03:50	1
trans-1,2-Dichloroethene	ND		3.0	0.39	ug/L			09/01/18 03:50	1
trans-1,3-Dichloropropene	ND		1.0	0.16	ug/L			09/01/18 03:50	1
Trichloroethene	ND		3.0	0.85	ug/L			09/01/18 03:50	1
Trichlorofluoromethane	ND		3.0	0.63	ug/L			09/01/18 03:50	1
Vinyl chloride	ND		1.0	0.22	ug/L			09/01/18 03:50	1

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*100*		*80 - 126*		*09/01/18 03:50*	*1*
4-Bromofluorobenzene (Surr)	*99*		*80 - 125*		*09/01/18 03:50*	*1*
Dibromofluoromethane (Surr)	*100*		*77 - 120*		*09/01/18 03:50*	*1*
Toluene-d8 (Surr)	*101*		*80 - 122*		*09/01/18 03:50*	*1*
Trifluorotoluene (Surr)	*101*		*80 - 120*		*09/01/18 03:50*	*1*

Lab Sample ID: LCS 580-283011/6
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Lab Control Sample
Prep Type: Total/NA

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,1,1,2-Tetrachloroethane	10.0	9.73		ug/L		97	79 - 120
1,1,1-Trichloroethane	10.0	9.80		ug/L		98	74 - 130
1,1,2,2-Tetrachloroethane	10.0	8.84		ug/L		88	65 - 130
1,1,2-Trichloroethane	10.0	8.95		ug/L		89	78 - 121
1,1-Dichloroethane	10.0	9.53		ug/L		95	70 - 129
1,1-Dichloroethene	10.0	9.69		ug/L		97	70 - 129
1,1-Dichloropropene	10.0	9.10		ug/L		91	75 - 125
1,2,3-Trichlorobenzene	10.0	7.98		ug/L		80	51 - 142
1,2,3-Trichloropropane	10.0	9.21		ug/L		92	76 - 124
1,2,4-Trichlorobenzene	10.0	8.64		ug/L		86	63 - 129
1,2,4-Trimethylbenzene	10.0	9.52		ug/L		95	75 - 121
1,2-Dibromo-3-Chloropropane	10.0	8.60	J	ug/L		86	65 - 133
1,2-Dibromoethane	10.0	8.63		ug/L		86	79 - 120
1,2-Dichlorobenzene	10.0	9.26		ug/L		93	80 - 120
1,2-Dichloroethane	10.0	9.13		ug/L		91	76 - 131
1,2-Dichloropropane	10.0	9.35		ug/L		94	72 - 126
1,3,5-Trimethylbenzene	10.0	9.52		ug/L		95	75 - 122
1,3-Dichlorobenzene	10.0	9.45		ug/L		95	80 - 120

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCS 580-283011/6
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Lab Control Sample
Prep Type: Total/NA

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,3-Dichloropropane	10.0	8.87		ug/L		89	79 - 120
1,4-Dichlorobenzene	10.0	9.36		ug/L		94	80 - 120
2,2-Dichloropropane	10.0	8.54		ug/L		85	62 - 140
2-Butanone	50.0	43.4		ug/L		87	65 - 135
2-Chlorotoluene	10.0	9.31		ug/L		93	80 - 120
2-Hexanone	50.0	42.6		ug/L		85	65 - 125
4-Chlorotoluene	10.0	9.55		ug/L		95	80 - 120
4-Isopropyltoluene	10.0	9.19		ug/L		92	77 - 120
4-Methyl-2-pentanone	50.0	43.4		ug/L		87	76 - 124
Acetone	50.0	48.3	J	ug/L		97	52 - 150
Benzene	10.0	9.49		ug/L		95	75 - 128
Bromobenzene	10.0	9.64		ug/L		96	75 - 120
Bromochloromethane	10.0	8.95		ug/L		89	78 - 128
Bromodichloromethane	10.0	9.51		ug/L		95	75 - 132
Bromoform	10.0	8.79		ug/L		88	61 - 132
Bromomethane	10.0	9.71		ug/L		97	66 - 125
Carbon disulfide	10.0	8.86		ug/L		89	69 - 122
Carbon tetrachloride	10.0	9.84		ug/L		98	72 - 139
Chlorobenzene	10.0	9.52		ug/L		95	80 - 120
Chloroethane	10.0	9.12		ug/L		91	65 - 132
Chloroform	10.0	9.90		ug/L		99	73 - 127
Chloromethane	10.0	8.61	J	ug/L		86	52 - 149
cis-1,2-Dichloroethene	10.0	9.83		ug/L		98	76 - 129
cis-1,3-Dichloropropene	10.0	8.95		ug/L		90	77 - 120
Dibromochloromethane	10.0	8.89		ug/L		89	71 - 120
Dibromomethane	10.0	8.74		ug/L		87	75 - 123
Dichlorodifluoromethane	10.0	8.47	J	ug/L		85	28 - 150
Ethylbenzene	10.0	9.43		ug/L		94	75 - 120
Hexachlorobutadiene	10.0	8.24		ug/L		82	65 - 125
Isopropylbenzene	10.0	9.69		ug/L		97	75 - 120
Methyl tert-butyl ether	10.0	8.80		ug/L		88	72 - 130
Methylene Chloride	10.0	10.4		ug/L		104	70 - 125
m-Xylene & p-Xylene	10.0	9.52		ug/L		95	75 - 120
Naphthalene	10.0	8.29		ug/L		83	50 - 144
n-Butylbenzene	10.0	9.14		ug/L		91	78 - 120
N-Propylbenzene	10.0	9.48		ug/L		95	73 - 124
o-Xylene	10.0	9.74		ug/L		97	74 - 120
sec-Butylbenzene	10.0	9.41		ug/L		94	78 - 125
Styrene	10.0	8.89		ug/L		89	76 - 121
t-Butylbenzene	10.0	9.81		ug/L		98	80 - 121
Tetrachloroethene	10.0	9.11		ug/L		91	76 - 120
Toluene	10.0	9.27		ug/L		93	75 - 120
trans-1,2-Dichloroethene	10.0	9.58		ug/L		96	72 - 124
trans-1,3-Dichloropropene	10.0	8.63		ug/L		86	73 - 122
Trichloroethene	10.0	9.80		ug/L		98	70 - 131
Trichlorofluoromethane	10.0	9.47		ug/L		95	64 - 136
Vinyl chloride	10.0	9.13		ug/L		91	65 - 130

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCS 580-283011/6
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Lab Control Sample
Prep Type: Total/NA

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*98*		*80 - 126*
4-Bromofluorobenzene (Surr)	*100*		*80 - 125*
Dibromofluoromethane (Surr)	*100*		*77 - 120*
Toluene-d8 (Surr)	*102*		*80 - 122*
Trifluorotoluene (Surr)	*102*		*80 - 120*

Lab Sample ID: LCSD 580-283011/7
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,1,1,2-Tetrachloroethane			10.0	9.65		ug/L		97	79 - 120	1	20
1,1,1-Trichloroethane			10.0	9.90		ug/L		99	74 - 130	1	18
1,1,2,2-Tetrachloroethane			10.0	8.19		ug/L		82	65 - 130	8	18
1,1,2-Trichloroethane			10.0	9.16		ug/L		92	78 - 121	2	14
1,1-Dichloroethane			10.0	9.75		ug/L		98	70 - 129	2	20
1,1-Dichloroethene			10.0	9.93		ug/L		99	70 - 129	2	27
1,1-Dichloropropene			10.0	9.50		ug/L		95	75 - 125	4	20
1,2,3-Trichlorobenzene			10.0	8.11		ug/L		81	51 - 142	2	17
1,2,3-Trichloropropane			10.0	8.60		ug/L		86	76 - 124	7	30
1,2,4-Trichlorobenzene			10.0	8.82		ug/L		88	63 - 129	2	22
1,2,4-Trimethylbenzene			10.0	9.22		ug/L		92	75 - 121	3	16
1,2-Dibromo-3-Chloropropane			10.0	8.54	J	ug/L		85	65 - 133	1	35
1,2-Dibromoethane			10.0	8.80		ug/L		88	79 - 120	2	26
1,2-Dichlorobenzene			10.0	9.16		ug/L		92	80 - 120	1	15
1,2-Dichloroethane			10.0	9.35		ug/L		94	76 - 131	2	11
1,2-Dichloropropane			10.0	9.29		ug/L		93	72 - 126	1	26
1,3,5-Trimethylbenzene			10.0	9.25		ug/L		92	75 - 122	3	14
1,3-Dichlorobenzene			10.0	9.12		ug/L		91	80 - 120	4	14
1,3-Dichloropropane			10.0	9.05		ug/L		91	79 - 120	2	35
1,4-Dichlorobenzene			10.0	9.31		ug/L		93	80 - 120	1	17
2,2-Dichloropropane			10.0	8.41		ug/L		84	62 - 140	2	35
2-Butanone			50.0	46.4		ug/L		93	65 - 135	7	35
2-Chlorotoluene			10.0	9.38		ug/L		94	80 - 120	1	15
2-Hexanone			50.0	43.2		ug/L		86	65 - 125	1	30
4-Chlorotoluene			10.0	9.37		ug/L		94	80 - 120	2	34
4-Isopropyltoluene			10.0	9.09		ug/L		91	77 - 120	1	13
4-Methyl-2-pentanone			50.0	44.2		ug/L		88	76 - 124	2	30
Acetone			50.0	49.9	J	ug/L		100	52 - 150	3	35
Benzene			10.0	9.57		ug/L		96	75 - 128	1	14
Bromobenzene			10.0	9.34		ug/L		93	75 - 120	3	13
Bromochloromethane			10.0	9.32		ug/L		93	78 - 128	4	35
Bromodichloromethane			10.0	9.62		ug/L		96	75 - 132	1	14
Bromoform			10.0	8.89		ug/L		89	61 - 132	1	20
Bromomethane			10.0	9.54		ug/L		95	66 - 125	2	35
Carbon disulfide			10.0	8.66		ug/L		87	69 - 122	2	20
Carbon tetrachloride			10.0	9.87		ug/L		99	72 - 139	0	19
Chlorobenzene			10.0	9.45		ug/L		95	80 - 120	1	15

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283011/7
Matrix: Water
Analysis Batch: 283011

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Chloroethane			10.0	9.18		ug/L		92	65 - 132	1	35
Chloroform			10.0	10.1		ug/L		101	73 - 127	2	15
Chloromethane			10.0	8.46	J	ug/L		85	52 - 149	2	35
cis-1,2-Dichloroethene			10.0	9.91		ug/L		99	76 - 129	1	15
cis-1,3-Dichloropropene			10.0	9.00		ug/L		90	77 - 120	1	12
Dibromochloromethane			10.0	9.03		ug/L		90	71 - 120	2	35
Dibromomethane			10.0	8.92		ug/L		89	75 - 123	2	22
Dichlorodifluoromethane			10.0	8.52	J	ug/L		85	28 - 150	1	35
Ethylbenzene			10.0	9.34		ug/L		93	75 - 120	1	14
Hexachlorobutadiene			10.0	8.10		ug/L		81	65 - 125	2	29
Isopropylbenzene			10.0	9.57		ug/L		96	75 - 120	1	20
Methyl tert-butyl ether			10.0	8.80		ug/L		88	72 - 130	0	18
Methylene Chloride			10.0	10.3		ug/L		103	70 - 125	1	29
m-Xylene & p-Xylene			10.0	9.57		ug/L		96	75 - 120	1	14
Naphthalene			10.0	8.40		ug/L		84	50 - 144	1	16
n-Butylbenzene			10.0	8.80		ug/L		88	78 - 120	4	14
N-Propylbenzene			10.0	9.03		ug/L		90	73 - 124	5	13
o-Xylene			10.0	9.58		ug/L		96	74 - 120	2	16
sec-Butylbenzene			10.0	9.19		ug/L		92	78 - 125	2	15
Styrene			10.0	9.08		ug/L		91	76 - 121	2	16
t-Butylbenzene			10.0	9.61		ug/L		96	80 - 121	2	14
Tetrachloroethene			10.0	9.09		ug/L		91	76 - 120	0	20
Toluene			10.0	9.36		ug/L		94	75 - 120	1	13
trans-1,2-Dichloroethene			10.0	9.78		ug/L		98	72 - 124	2	21
trans-1,3-Dichloropropene			10.0	8.67		ug/L		87	73 - 122	1	13
Trichloroethene			10.0	10.3		ug/L		103	70 - 131	5	15
Trichlorofluoromethane			10.0	9.55		ug/L		95	64 - 136	1	35
Vinyl chloride			10.0	9.19		ug/L		92	65 - 130	1	35

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*100*		*80 - 126*
4-Bromofluorobenzene (Surr)	*103*		*80 - 125*
Dibromofluoromethane (Surr)	*100*		*77 - 120*
Toluene-d8 (Surr)	*102*		*80 - 122*
Trifluorotoluene (Surr)	*102*		*80 - 120*

Lab Sample ID: MB 580-283200/1-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 283200

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		40	11	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1,1-Trichloroethane	ND		40	9.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1,2,2-Tetrachloroethane	ND		20	7.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1,2-Trichloroethane	ND		20	7.4	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1-Dichloroethane	ND		40	4.2	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1-Dichloroethene	ND		40	12	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,1-Dichloropropene	ND		40	5.3	ug/Kg		09/04/18 18:30	09/04/18 21:47	1

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: MB 580-283200/1-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 283200

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,2,3-Trichlorobenzene	ND		150	32	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2,3-Trichloropropane	ND		40	12	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2,4-Trichlorobenzene	ND		60	15	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2,4-Trimethylbenzene	ND		40	14	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2-Dibromo-3-Chloropropane	ND		250	40	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2-Dibromoethane	ND		20	3.8	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2-Dichlorobenzene	ND		40	8.7	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2-Dichloroethane	ND		20	5.5	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,2-Dichloropropane	ND		20	6.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,3,5-Trimethylbenzene	ND		40	7.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,3-Dichlorobenzene	ND		60	13	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,3-Dichloropropane	ND		60	14	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
1,4-Dichlorobenzene	ND		60	11	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
2,2-Dichloropropane	ND		40	12	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
2-Butanone	ND		600	190	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
2-Chlorotoluene	ND		40	8.8	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
2-Hexanone	ND		100	36	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
4-Chlorotoluene	ND		40	9.8	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
4-Isopropyltoluene	ND		40	10	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
4-Methyl-2-pentanone	ND		400	81	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Acetone	ND		800	170	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Benzene	ND		30	7.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Bromobenzene	ND		100	17	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Bromochloromethane	ND		40	6.2	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Bromodichloromethane	ND		60	13	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Bromoform	ND		200	26	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Bromomethane	ND		200	13	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Carbon disulfide	ND		60	12	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Carbon tetrachloride	ND		20	3.8	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Chlorobenzene	ND		40	9.8	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Chloroethane	ND		400	54	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Chloroform	ND		40	4.2	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Chloromethane	ND		100	10	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
cis-1,2-Dichloroethene	ND		60	13	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
cis-1,3-Dichloropropene	ND		20	4.0	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Dibromochloromethane	ND		40	11	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Dibromomethane	ND		60	7.4	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Dichlorodifluoromethane	ND		200	46	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Ethylbenzene	ND		40	9.1	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Hexachlorobutadiene	ND		150	33	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Isopropylbenzene	ND		40	8.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Methyl tert-butyl ether	ND		40	6.0	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Methylene Chloride	ND		250	65	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
m-Xylene & p-Xylene	ND		200	15	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Naphthalene	ND		100	28	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
n-Butylbenzene	ND		150	25	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
N-Propylbenzene	ND		40	6.9	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
o-Xylene	ND		60	13	ug/Kg		09/04/18 18:30	09/04/18 21:47	1

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: MB 580-283200/1-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 283200

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
sec-Butylbenzene	ND		40	8.6	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Styrene	ND		40	6.1	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
t-Butylbenzene	ND		40	7.7	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Tetrachloroethene	ND		40	5.3	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Toluene	ND		150	14	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
trans-1,2-Dichloroethene	ND		60	15	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
trans-1,3-Dichloropropene	ND		40	7.0	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Trichloroethene	ND		60	22	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Trichlorofluoromethane	ND		200	11	ug/Kg		09/04/18 18:30	09/04/18 21:47	1
Vinyl chloride	ND		150	26	ug/Kg		09/04/18 18:30	09/04/18 21:47	1

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*89*		*80 - 121*	*09/04/18 18:30*	*09/04/18 21:47*	*1*
4-Bromofluorobenzene (Surr)	*104*		*80 - 120*	*09/04/18 18:30*	*09/04/18 21:47*	*1*
Dibromofluoromethane (Surr)	*96*		*80 - 120*	*09/04/18 18:30*	*09/04/18 21:47*	*1*
Toluene-d8 (Surr)	*100*		*80 - 120*	*09/04/18 18:30*	*09/04/18 21:47*	*1*
Trifluorotoluene (Surr)	*105*		*80 - 120*	*09/04/18 18:30*	*09/04/18 21:47*	*1*

Lab Sample ID: LCS 580-283200/2-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 283200

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,1,1,2-Tetrachloroethane	800	793		ug/Kg		99	79 - 128
1,1,1-Trichloroethane	800	851		ug/Kg		106	69 - 144
1,1,2,2-Tetrachloroethane	800	639		ug/Kg		80	74 - 120
1,1,2-Trichloroethane	800	734		ug/Kg		92	80 - 123
1,1-Dichloroethane	800	786		ug/Kg		98	70 - 141
1,1-Dichloroethene	800	828		ug/Kg		103	68 - 137
1,1-Dichloropropene	800	858		ug/Kg		107	76 - 141
1,2,3-Trichlorobenzene	800	725		ug/Kg		91	71 - 129
1,2,3-Trichloropropane	800	687		ug/Kg		86	70 - 127
1,2,4-Trichlorobenzene	800	762		ug/Kg		95	68 - 131
1,2,4-Trimethylbenzene	800	798		ug/Kg		100	73 - 127
1,2-Dibromo-3-Chloropropane	800	641		ug/Kg		80	53 - 135
1,2-Dibromoethane	800	725		ug/Kg		91	77 - 123
1,2-Dichlorobenzene	800	797		ug/Kg		100	78 - 120
1,2-Dichloroethane	800	721		ug/Kg		90	68 - 132
1,2-Dichloropropane	800	766		ug/Kg		96	75 - 136
1,3,5-Trimethylbenzene	800	825		ug/Kg		103	72 - 128
1,3-Dichlorobenzene	800	822		ug/Kg		103	78 - 122
1,3-Dichloropropane	800	693		ug/Kg		87	80 - 120
1,4-Dichlorobenzene	800	836		ug/Kg		105	77 - 123
2,2-Dichloropropane	800	850		ug/Kg		106	62 - 150
2-Butanone	4000	2590		ug/Kg		65	63 - 138
2-Chlorotoluene	800	859		ug/Kg		107	77 - 127
2-Hexanone	4000	2950		ug/Kg		74	70 - 134
4-Chlorotoluene	800	849		ug/Kg		106	78 - 126

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCS 580-283200/2-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 283200

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
4-Isopropyltoluene	800	870		ug/Kg		109	71 - 129
4-Methyl-2-pentanone	4000	3060		ug/Kg		76	73 - 125
Acetone	4000	2120		ug/Kg		53	39 - 150
Benzene	800	821		ug/Kg		103	79 - 135
Bromobenzene	800	796		ug/Kg		100	78 - 126
Bromochloromethane	800	827		ug/Kg		103	76 - 131
Bromodichloromethane	800	748		ug/Kg		94	73 - 132
Bromoform	800	707		ug/Kg		88	65 - 134
Bromomethane	800	815		ug/Kg		102	66 - 133
Carbon disulfide	800	761		ug/Kg		95	68 - 131
Carbon tetrachloride	800	887		ug/Kg		111	66 - 150
Chlorobenzene	800	833		ug/Kg		104	80 - 123
Chloroethane	800	729		ug/Kg		91	67 - 139
Chloroform	800	801		ug/Kg		100	74 - 133
Chloromethane	800	650		ug/Kg		81	53 - 145
cis-1,2-Dichloroethene	800	884		ug/Kg		110	74 - 129
cis-1,3-Dichloropropene	800	702		ug/Kg		88	80 - 122
Dibromochloromethane	800	741		ug/Kg		93	75 - 125
Dibromomethane	800	802		ug/Kg		100	72 - 130
Dichlorodifluoromethane	800	589		ug/Kg		74	26 - 145
Ethylbenzene	800	840		ug/Kg		105	80 - 127
Hexachlorobutadiene	800	893		ug/Kg		112	65 - 136
Isopropylbenzene	800	849		ug/Kg		106	80 - 128
Methyl tert-butyl ether	800	724		ug/Kg		90	75 - 126
Methylene Chloride	800	792		ug/Kg		99	66 - 141
m-Xylene & p-Xylene	800	818		ug/Kg		102	80 - 128
Naphthalene	800	659		ug/Kg		82	67 - 124
n-Butylbenzene	800	856		ug/Kg		107	77 - 130
N-Propylbenzene	800	865		ug/Kg		108	74 - 127
o-Xylene	800	806		ug/Kg		101	80 - 125
sec-Butylbenzene	800	868		ug/Kg		108	77 - 129
Styrene	800	823		ug/Kg		103	79 - 129
t-Butylbenzene	800	859		ug/Kg		107	79 - 127
Tetrachloroethene	800	928		ug/Kg		116	71 - 136
Toluene	800	786		ug/Kg		98	80 - 125
trans-1,2-Dichloroethene	800	845		ug/Kg		106	71 - 135
trans-1,3-Dichloropropene	800	732		ug/Kg		92	80 - 121
Trichloroethene	800	895		ug/Kg		112	69 - 144
Trichlorofluoromethane	800	871		ug/Kg		109	73 - 143
Vinyl chloride	800	829		ug/Kg		104	52 - 150

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*89*		*80 - 121*
4-Bromofluorobenzene (Surr)	*102*		*80 - 120*
Dibromofluoromethane (Surr)	*100*		*80 - 120*
Toluene-d8 (Surr)	*96*		*80 - 120*
Trifluorotoluene (Surr)	*107*		*80 - 120*

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283200/3-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 283200

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,1,1,2-Tetrachloroethane	800	817		ug/Kg		102	79 - 128	3	11
1,1,1-Trichloroethane	800	866		ug/Kg		108	69 - 144	2	14
1,1,2,2-Tetrachloroethane	800	699		ug/Kg		87	74 - 120	9	18
1,1,2-Trichloroethane	800	775		ug/Kg		97	80 - 123	5	15
1,1-Dichloroethane	800	799		ug/Kg		100	70 - 141	2	13
1,1-Dichloroethene	800	867		ug/Kg		108	68 - 137	5	17
1,1-Dichloropropene	800	857		ug/Kg		107	76 - 141	0	11
1,2,3-Trichlorobenzene	800	820		ug/Kg		103	71 - 129	12	18
1,2,3-Trichloropropane	800	716		ug/Kg		89	70 - 127	4	16
1,2,4-Trichlorobenzene	800	828		ug/Kg		103	68 - 131	8	16
1,2,4-Trimethylbenzene	800	810		ug/Kg		101	73 - 127	1	12
1,2-Dibromo-3-Chloropropane	800	729		ug/Kg		91	53 - 135	13	20
1,2-Dibromoethane	800	786		ug/Kg		98	77 - 123	8	11
1,2-Dichlorobenzene	800	824		ug/Kg		103	78 - 120	3	12
1,2-Dichloroethane	800	775		ug/Kg		97	68 - 132	7	11
1,2-Dichloropropane	800	782		ug/Kg		98	75 - 136	2	10
1,3,5-Trimethylbenzene	800	839		ug/Kg		105	72 - 128	2	16
1,3-Dichlorobenzene	800	834		ug/Kg		104	78 - 122	1	12
1,3-Dichloropropane	800	752		ug/Kg		94	80 - 120	8	18
1,4-Dichlorobenzene	800	847		ug/Kg		106	77 - 123	1	12
2,2-Dichloropropane	800	853		ug/Kg		107	62 - 150	0	20
2-Butanone	4000	3410		ug/Kg		85	63 - 138	27	31
2-Chlorotoluene	800	858		ug/Kg		107	77 - 127	0	16
2-Hexanone	4000	3440		ug/Kg		86	70 - 134	15	21
4-Chlorotoluene	800	853		ug/Kg		107	78 - 126	0	16
4-Isopropyltoluene	800	874		ug/Kg		109	71 - 129	0	11
4-Methyl-2-pentanone	4000	3560		ug/Kg		89	73 - 125	15	20
Acetone	4000	2910		ug/Kg		73	39 - 150	32	33
Benzene	800	831		ug/Kg		104	79 - 135	1	15
Bromobenzene	800	809		ug/Kg		101	78 - 126	2	12
Bromochloromethane	800	861		ug/Kg		108	76 - 131	4	15
Bromodichloromethane	800	794		ug/Kg		99	73 - 132	6	10
Bromoform	800	763		ug/Kg		95	65 - 134	8	17
Bromomethane	800	748		ug/Kg		93	66 - 133	9	22
Carbon disulfide	800	807		ug/Kg		101	68 - 131	6	13
Carbon tetrachloride	800	902		ug/Kg		113	66 - 150	2	12
Chlorobenzene	800	847		ug/Kg		106	80 - 123	2	10
Chloroethane	800	655		ug/Kg		82	67 - 139	11	22
Chloroform	800	810		ug/Kg		101	74 - 133	1	13
Chloromethane	800	609		ug/Kg		76	53 - 145	7	18
cis-1,2-Dichloroethene	800	889		ug/Kg		111	74 - 129	1	14
cis-1,3-Dichloropropene	800	729		ug/Kg		91	80 - 122	4	16
Dibromochloromethane	800	793		ug/Kg		99	75 - 125	7	11
Dibromomethane	800	860		ug/Kg		108	72 - 130	7	14
Dichlorodifluoromethane	800	566		ug/Kg		71	26 - 145	4	23
Ethylbenzene	800	849		ug/Kg		106	80 - 127	1	16
Hexachlorobutadiene	800	898		ug/Kg		112	65 - 136	1	19
Isopropylbenzene	800	876		ug/Kg		109	80 - 128	3	17

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283200/3-A
Matrix: Solid
Analysis Batch: 283203

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 283200

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Methyl tert-butyl ether			800	764		ug/Kg		96	75 - 126	5	15
Methylene Chloride			800	829		ug/Kg		104	66 - 141	4	17
m-Xylene & p-Xylene			800	832		ug/Kg		104	80 - 128	2	13
Naphthalene			800	775		ug/Kg		97	67 - 124	16	17
n-Butylbenzene			800	873		ug/Kg		109	77 - 130	2	12
N-Propylbenzene			800	842		ug/Kg		105	74 - 127	3	17
o-Xylene			800	821		ug/Kg		103	80 - 125	2	14
sec-Butylbenzene			800	874		ug/Kg		109	77 - 129	1	12
Styrene			800	858		ug/Kg		107	79 - 129	4	15
t-Butylbenzene			800	868		ug/Kg		109	79 - 127	1	13
Tetrachloroethene			800	941		ug/Kg		118	71 - 136	1	16
Toluene			800	807		ug/Kg		101	80 - 125	3	16
trans-1,2-Dichloroethene			800	834		ug/Kg		104	71 - 135	1	16
trans-1,3-Dichloropropene			800	750		ug/Kg		94	80 - 121	2	17
Trichloroethene			800	919		ug/Kg		115	69 - 144	3	10
Trichlorofluoromethane			800	898		ug/Kg		112	73 - 143	3	17
Vinyl chloride			800	773		ug/Kg		97	52 - 150	7	37

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*92*		*80 - 121*
4-Bromofluorobenzene (Surr)	*101*		*80 - 120*
Dibromofluoromethane (Surr)	*100*		*80 - 120*
Toluene-d8 (Surr)	*98*		*80 - 120*
Trifluorotoluene (Surr)	*106*		*80 - 120*

Lab Sample ID: MB 580-283436/1-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Method Blank
Prep Type: SPLP

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1,2-Tetrachloroethane	ND		200	18	ug/L			09/08/18 10:11	100
1,1,1-Trichloroethane	ND		300	14	ug/L			09/08/18 10:11	100
1,1,2,2-Tetrachloroethane	ND		300	52	ug/L			09/08/18 10:11	100
1,1-Dichloroethane	ND		200	22	ug/L			09/08/18 10:11	100
1,1-Dichloroethene	ND		400	78	ug/L			09/08/18 10:11	100
1,1-Dichloropropene	ND		300	29	ug/L			09/08/18 10:11	100
1,2,3-Trichlorobenzene	ND		500	46	ug/L			09/08/18 10:11	100
1,2,3-Trichloropropane	ND		200	41	ug/L			09/08/18 10:11	100
1,2,4-Trichlorobenzene	ND		200	33	ug/L			09/08/18 10:11	100
1,2,4-Trimethylbenzene	ND		300	61	ug/L			09/08/18 10:11	100
1,2-Dibromo-3-Chloropropane	ND		1000	180	ug/L			09/08/18 10:11	100
1,2-Dibromoethane	ND		200	40	ug/L			09/08/18 10:11	100
1,2-Dichlorobenzene	ND		200	46	ug/L			09/08/18 10:11	100
1,2-Dichloroethane	ND		200	53	ug/L			09/08/18 10:11	100
1,2-Dichloropropane	ND		100	18	ug/L			09/08/18 10:11	100
1,3,5-Trimethylbenzene	ND		300	55	ug/L			09/08/18 10:11	100
1,3-Dichlorobenzene	ND		200	18	ug/L			09/08/18 10:11	100
1,3-Dichloropropane	ND		200	35	ug/L			09/08/18 10:11	100

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: MB 580-283436/1-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Method Blank
Prep Type: SPLP

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,4-Dichlorobenzene	ND		400	98	ug/L			09/08/18 10:11	100
2,2-Dichloropropane	ND		300	32	ug/L			09/08/18 10:11	100
2-Butanone	ND		2000	470	ug/L			09/08/18 10:11	100
2-Chlorotoluene	ND		300	51	ug/L			09/08/18 10:11	100
2-Hexanone	ND		2000	400	ug/L			09/08/18 10:11	100
4-Chlorotoluene	ND		200	51	ug/L			09/08/18 10:11	100
4-Isopropyltoluene	ND		300	28	ug/L			09/08/18 10:11	100
4-Methyl-2-pentanone	ND		1500	250	ug/L			09/08/18 10:11	100
Acetone	ND		5000	780	ug/L			09/08/18 10:11	100
Benzene	ND		300	53	ug/L			09/08/18 10:11	100
Bromobenzene	ND		200	18	ug/L			09/08/18 10:11	100
Bromochloromethane	ND		200	29	ug/L			09/08/18 10:11	100
Bromodichloromethane	ND		200	14	ug/L			09/08/18 10:11	100
Bromoform	ND		300	56	ug/L			09/08/18 10:11	100
Bromomethane	ND		600	110	ug/L			09/08/18 10:11	100
Carbon disulfide	ND		300	53	ug/L			09/08/18 10:11	100
Carbon tetrachloride	ND		300	30	ug/L			09/08/18 10:11	100
Chlorobenzene	ND		200	44	ug/L			09/08/18 10:11	100
Chloroethane	ND		500	110	ug/L			09/08/18 10:11	100
Chloroform	ND		500	50	ug/L			09/08/18 10:11	100
Chloromethane	ND		2000	540	ug/L			09/08/18 10:11	100
cis-1,2-Dichloroethene	ND		300	69	ug/L			09/08/18 10:11	100
cis-1,3-Dichloropropene	ND		100	20	ug/L			09/08/18 10:11	100
Dibromochloromethane	ND		100	20	ug/L			09/08/18 10:11	100
Dichlorodifluoromethane	ND		1000	230	ug/L			09/08/18 10:11	100
Ethylbenzene	ND		300	50	ug/L			09/08/18 10:11	100
Hexachlorobutadiene	ND		600	79	ug/L			09/08/18 10:11	100
Isopropylbenzene	ND		200	51	ug/L			09/08/18 10:11	100
Methyl tert-butyl ether	ND		200	44	ug/L			09/08/18 10:11	100
Methylene Chloride	ND		500	140	ug/L			09/08/18 10:11	100
m-Xylene & p-Xylene	ND		300	75	ug/L			09/08/18 10:11	100
Naphthalene	ND		400	93	ug/L			09/08/18 10:11	100
n-Butylbenzene	ND		300	44	ug/L			09/08/18 10:11	100
N-Propylbenzene	ND		300	50	ug/L			09/08/18 10:11	100
o-Xylene	ND		200	15	ug/L			09/08/18 10:11	100
sec-Butylbenzene	ND		300	49	ug/L			09/08/18 10:11	100
Styrene	ND		500	51	ug/L			09/08/18 10:11	100
t-Butylbenzene	ND		300	58	ug/L			09/08/18 10:11	100
Tetrachloroethene	ND		300	41	ug/L			09/08/18 10:11	100
Toluene	ND		200	39	ug/L			09/08/18 10:11	100
trans-1,2-Dichloroethene	ND		300	39	ug/L			09/08/18 10:11	100
trans-1,3-Dichloropropene	ND		100	16	ug/L			09/08/18 10:11	100
Trichloroethene	ND		300	85	ug/L			09/08/18 10:11	100
Trichlorofluoromethane	ND		300	63	ug/L			09/08/18 10:11	100
Vinyl chloride	ND		100	22	ug/L			09/08/18 10:11	100

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*90*		*80 - 126*		*09/08/18 10:11*	*100*

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: MB 580-283436/1-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Method Blank
Prep Type: SPLP

Surrogate	MB %Recovery	MB Qualifier	Limits	Prepared	Analyzed	Dil Fac
4-Bromofluorobenzene (Surr)	97		80 - 125		09/08/18 10:11	100
Dibromofluoromethane (Surr)	96		77 - 120		09/08/18 10:11	100
Toluene-d8 (Surr)	103		80 - 122		09/08/18 10:11	100
Trifluorotoluene (Surr)	104		80 - 120		09/08/18 10:11	100

Lab Sample ID: LCS 580-283436/2-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Lab Control Sample
Prep Type: SPLP

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,1,1,2-Tetrachloroethane	1000	899		ug/L		90	79 - 120
1,1,1-Trichloroethane	1000	984		ug/L		98	74 - 130
1,1,2,2-Tetrachloroethane	1000	774		ug/L		77	65 - 130
1,1-Dichloroethane	1000	938		ug/L		94	70 - 129
1,1-Dichloroethene	1000	953		ug/L		95	70 - 129
1,1-Dichloropropene	1000	934		ug/L		93	75 - 125
1,2,3-Trichlorobenzene	1000	680		ug/L		68	51 - 142
1,2,3-Trichloropropane	1000	791		ug/L		79	76 - 124
1,2,4-Trichlorobenzene	1000	738		ug/L		74	63 - 129
1,2,4-Trimethylbenzene	1000	963		ug/L		96	75 - 121
1,2-Dibromo-3-Chloropropane	1000	650	J	ug/L		65	65 - 133
1,2-Dichlorobenzene	1000	865		ug/L		87	80 - 120
1,2-Dichloroethane	1000	818		ug/L		82	76 - 131
1,2-Dichloropropane	1000	873		ug/L		87	72 - 126
1,3,5-Trimethylbenzene	1000	970		ug/L		97	75 - 122
1,3-Dichlorobenzene	1000	877		ug/L		88	80 - 120
1,3-Dichloropropane	1000	782	*	ug/L		78	79 - 120
1,4-Dichlorobenzene	1000	910		ug/L		91	80 - 120
2,2-Dichloropropane	1000	1020		ug/L		102	62 - 140
2-Butanone	5000	3730		ug/L		75	65 - 135
2-Chlorotoluene	1000	979		ug/L		98	80 - 120
2-Hexanone	5000	3550		ug/L		71	65 - 125
4-Chlorotoluene	1000	984		ug/L		98	80 - 120
4-Isopropyltoluene	1000	969		ug/L		97	77 - 120
Acetone	5000	4010	J	ug/L		80	52 - 150
Benzene	1000	932		ug/L		93	75 - 128
Bromobenzene	1000	939		ug/L		94	75 - 120
Bromochloromethane	1000	814		ug/L		81	78 - 128
Bromodichloromethane	1000	884		ug/L		88	75 - 132
Bromoform	1000	732		ug/L		73	61 - 132
Bromomethane	1000	911		ug/L		91	66 - 125
Carbon disulfide	1000	828		ug/L		83	69 - 122
Carbon tetrachloride	1000	947		ug/L		95	72 - 139
Chlorobenzene	1000	934		ug/L		93	80 - 120
Chloroethane	1000	857		ug/L		86	65 - 132
Chloroform	1000	970		ug/L		97	73 - 127
Chloromethane	1000	804	J	ug/L		80	52 - 149
cis-1,2-Dichloroethene	1000	956		ug/L		96	76 - 129

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCS 580-283436/2-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Lab Control Sample
Prep Type: SPLP

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits		
cis-1,3-Dichloropropene			1000	897		ug/L		90	77 - 120		
Dibromochloromethane			1000	778		ug/L		78	71 - 120		
Dichlorodifluoromethane			1000	705	J	ug/L		70	28 - 150		
Ethylbenzene			1000	958		ug/L		96	75 - 120		
Hexachlorobutadiene			1000	835		ug/L		84	65 - 125		
Isopropylbenzene			1000	943		ug/L		94	75 - 120		
Methyl tert-butyl ether			1000	752		ug/L		75	72 - 130		
Methylene Chloride			1000	899		ug/L		90	70 - 125		
m-Xylene & p-Xylene			1000	947		ug/L		95	75 - 120		
Naphthalene			1000	648		ug/L		65	50 - 144		
n-Butylbenzene			1000	961		ug/L		96	78 - 120		
N-Propylbenzene			1000	1010		ug/L		101	73 - 124		
o-Xylene			1000	927		ug/L		93	74 - 120		
sec-Butylbenzene			1000	964		ug/L		96	78 - 125		
Styrene			1000	849		ug/L		85	76 - 121		
t-Butylbenzene			1000	1010		ug/L		101	80 - 121		
Tetrachloroethene			1000	925		ug/L		92	76 - 120		
Toluene			1000	925		ug/L		92	75 - 120		
trans-1,2-Dichloroethene			1000	930		ug/L		93	72 - 124		
trans-1,3-Dichloropropene			1000	789		ug/L		79	73 - 122		
Trichloroethene			1000	936		ug/L		94	70 - 131		
Trichlorofluoromethane			1000	901		ug/L		90	64 - 136		
Vinyl chloride			1000	833		ug/L		83	65 - 130		

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*88*		*80 - 126*
4-Bromofluorobenzene (Surr)	*96*		*80 - 125*
Dibromofluoromethane (Surr)	*96*		*77 - 120*
Toluene-d8 (Surr)	*103*		*80 - 122*
Trifluorotoluene (Surr)	*104*		*80 - 120*

Lab Sample ID: LCSD 580-283436/3-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,1,1,2-Tetrachloroethane			1000	908		ug/L		91	79 - 120	1	20
1,1,1-Trichloroethane			1000	960		ug/L		96	74 - 130	2	18
1,1,2,2-Tetrachloroethane			1000	789		ug/L		79	65 - 130	2	18
1,1-Dichloroethane			1000	934		ug/L		93	70 - 129	0	20
1,1-Dichloroethene			1000	944		ug/L		94	70 - 129	1	27
1,1-Dichloropropene			1000	931		ug/L		93	75 - 125	0	20
1,2,3-Trichlorobenzene			1000	732		ug/L		73	51 - 142	7	17
1,2,3-Trichloropropane			1000	796		ug/L		80	76 - 124	1	30
1,2,4-Trichlorobenzene			1000	755		ug/L		76	63 - 129	2	22
1,2,4-Trimethylbenzene			1000	974		ug/L		97	75 - 121	1	16
1,2-Dibromo-3-Chloropropane			1000	677	J	ug/L		68	65 - 133	4	35
1,2-Dichlorobenzene			1000	872		ug/L		87	80 - 120	1	15

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283436/3-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,2-Dichloroethane	1000	838		ug/L		84	76 - 131	2	11
1,2-Dichloropropane	1000	885		ug/L		89	72 - 126	1	26
1,3,5-Trimethylbenzene	1000	985		ug/L		98	75 - 122	2	14
1,3-Dichlorobenzene	1000	891		ug/L		89	80 - 120	2	14
1,3-Dichloropropane	1000	799		ug/L		80	79 - 120	2	35
1,4-Dichlorobenzene	1000	937		ug/L		94	80 - 120	3	17
2,2-Dichloropropane	1000	994		ug/L		99	62 - 140	2	35
2-Butanone	5000	3970		ug/L		79	65 - 135	6	35
2-Chlorotoluene	1000	980		ug/L		98	80 - 120	0	15
2-Hexanone	5000	3810		ug/L		76	65 - 125	7	30
4-Chlorotoluene	1000	1010		ug/L		101	80 - 120	3	34
4-Isopropyltoluene	1000	979		ug/L		98	77 - 120	1	13
Acetone	5000	4420	J	ug/L		88	52 - 150	10	35
Benzene	1000	930		ug/L		93	75 - 128	0	14
Bromobenzene	1000	949		ug/L		95	75 - 120	1	13
Bromochloromethane	1000	826		ug/L		83	78 - 128	1	35
Bromodichloromethane	1000	849		ug/L		85	75 - 132	4	14
Bromoform	1000	769		ug/L		77	61 - 132	5	20
Bromomethane	1000	866		ug/L		87	66 - 125	5	35
Carbon disulfide	1000	830		ug/L		83	69 - 122	0	20
Carbon tetrachloride	1000	961		ug/L		96	72 - 139	2	19
Chlorobenzene	1000	920		ug/L		92	80 - 120	1	15
Chloroethane	1000	856		ug/L		86	65 - 132	0	35
Chloroform	1000	965		ug/L		96	73 - 127	1	15
Chloromethane	1000	810	J	ug/L		81	52 - 149	1	35
cis-1,2-Dichloroethene	1000	913		ug/L		91	76 - 129	5	15
cis-1,3-Dichloropropene	1000	924		ug/L		92	77 - 120	3	12
Dibromochloromethane	1000	809		ug/L		81	71 - 120	4	35
Dichlorodifluoromethane	1000	736	J	ug/L		74	28 - 150	4	35
Ethylbenzene	1000	921		ug/L		92	75 - 120	4	14
Hexachlorobutadiene	1000	859		ug/L		86	65 - 125	3	29
Isopropylbenzene	1000	945		ug/L		95	75 - 120	0	20
Methyl tert-butyl ether	1000	755		ug/L		75	72 - 130	0	18
Methylene Chloride	1000	897		ug/L		90	70 - 125	0	29
m-Xylene & p-Xylene	1000	944		ug/L		94	75 - 120	0	14
Naphthalene	1000	713		ug/L		71	50 - 144	10	16
n-Butylbenzene	1000	959		ug/L		96	78 - 120	0	14
N-Propylbenzene	1000	1020		ug/L		102	73 - 124	1	13
o-Xylene	1000	946		ug/L		95	74 - 120	2	16
sec-Butylbenzene	1000	971		ug/L		97	78 - 125	1	15
Styrene	1000	863		ug/L		86	76 - 121	2	16
t-Butylbenzene	1000	1020		ug/L		102	80 - 121	1	14
Tetrachloroethene	1000	930		ug/L		93	76 - 120	1	20
Toluene	1000	944		ug/L		94	75 - 120	2	13
trans-1,2-Dichloroethene	1000	962		ug/L		96	72 - 124	3	21
trans-1,3-Dichloropropene	1000	801		ug/L		80	73 - 122	2	13
Trichloroethene	1000	923		ug/L		92	70 - 131	1	15
Trichlorofluoromethane	1000	895		ug/L		90	64 - 136	1	35

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283436/3-A
Matrix: Solid
Analysis Batch: 283529

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Vinyl chloride			1000	849		ug/L		85	65 - 130	2	35

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*90*		*80 - 126*
4-Bromofluorobenzene (Surr)	*97*		*80 - 125*
Dibromofluoromethane (Surr)	*97*		*77 - 120*
Toluene-d8 (Surr)	*102*		*80 - 122*
Trifluorotoluene (Surr)	*103*		*80 - 120*

Lab Sample ID: MB 580-283436/1-A
Matrix: Solid
Analysis Batch: 284209

Client Sample ID: Method Blank
Prep Type: SPLP

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,2-Trichloroethane	ND		100	24	ug/L			09/18/18 14:26	100
1,2-Dibromoethane	ND		200	40	ug/L			09/18/18 14:26	100
4-Methyl-2-pentanone	ND		1500	250	ug/L			09/18/18 14:26	100
Dibromomethane	ND		200	34	ug/L			09/18/18 14:26	100

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
1,2-Dichloroethane-d4 (Surr)	*95*		*80 - 126*		*09/18/18 14:26*	*100*
4-Bromofluorobenzene (Surr)	*98*		*80 - 125*		*09/18/18 14:26*	*100*
Dibromofluoromethane (Surr)	*96*		*77 - 120*		*09/18/18 14:26*	*100*
Toluene-d8 (Surr)	*106*		*80 - 122*		*09/18/18 14:26*	*100*
Trifluorotoluene (Surr)	*102*		*80 - 120*		*09/18/18 14:26*	*100*

Lab Sample ID: LCS 580-283436/2-A
Matrix: Solid
Analysis Batch: 284209

Client Sample ID: Lab Control Sample
Prep Type: SPLP

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,1,2-Trichloroethane			1000	1110		ug/L		111	78 - 121
1,2-Dibromoethane			1000	990		ug/L		99	79 - 120
4-Methyl-2-pentanone			5000	5150		ug/L		103	76 - 124
Dibromomethane			1000	1000		ug/L		100	75 - 123

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*101*		*80 - 126*
4-Bromofluorobenzene (Surr)	*103*		*80 - 125*
Dibromofluoromethane (Surr)	*96*		*77 - 120*
Toluene-d8 (Surr)	*101*		*80 - 122*
Trifluorotoluene (Surr)	*104*		*80 - 120*

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Lab Sample ID: LCSD 580-283436/3-A
Matrix: Solid
Analysis Batch: 284209

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,1,2-Trichloroethane			1000	986		ug/L		99	78 - 121	11	14
1,2-Dibromoethane			1000	1020		ug/L		102	79 - 120	3	26
4-Methyl-2-pentanone			5000	4930		ug/L		99	76 - 124	4	30
Dibromomethane			1000	962		ug/L		96	75 - 123	4	22

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
1,2-Dichloroethane-d4 (Surr)	*97*		*80 - 126*
4-Bromofluorobenzene (Surr)	*97*		*80 - 125*
Dibromofluoromethane (Surr)	*100*		*77 - 120*
Toluene-d8 (Surr)	*104*		*80 - 122*
Trifluorotoluene (Surr)	*99*		*80 - 120*

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Lab Sample ID: MB 580-282779/1-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 282779

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,3-Dichlorobenzene	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
1,4-Dichlorobenzene	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 19:09	1
1,2-Dichlorobenzene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Chlorophenol	ND		0.60	0.22	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Methylphenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4-Dimethylphenol	ND		4.0	0.83	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Nitrophenol	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 19:09	1
3 & 4 Methylphenol	ND		0.80	0.18	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4-Dichlorophenol	ND		4.0	0.53	ug/L		08/29/18 12:59	09/04/18 19:09	1
1,2,4-Trichlorobenzene	ND		0.40	0.040	ug/L		08/29/18 12:59	09/04/18 19:09	1
4-Chloroaniline	ND		10	2.1	ug/L		08/29/18 12:59	09/04/18 19:09	1
4-Chloro-3-methylphenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Methylnaphthalene	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4,6-Trichlorophenol	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4,5-Trichlorophenol	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Chloronaphthalene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 19:09	1
2-Nitroaniline	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Acenaphthylene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,6-Dinitrotoluene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
3-Nitroaniline	ND		3.0	0.73	ug/L		08/29/18 12:59	09/04/18 19:09	1
Acenaphthene	ND		0.40	0.080	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzoic acid	ND		4.0	0.85	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4-Dinitrophenol	ND		5.0	1.0	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzyl alcohol	ND		3.0	0.69	ug/L		08/29/18 12:59	09/04/18 19:09	1
4-Nitrophenol	ND		15	1.8	ug/L		08/29/18 12:59	09/04/18 19:09	1
Bis(2-chloroethoxy)methane	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Bis(2-chloroethyl)ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
2,4-Dinitrotoluene	ND		1.0	0.14	ug/L		08/29/18 12:59	09/04/18 19:09	1

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: MB 580-282779/1-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 282779

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4-Chlorophenyl phenyl ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
4-Nitroaniline	ND		2.0	0.29	ug/L		08/29/18 12:59	09/04/18 19:09	1
4,6-Dinitro-2-methylphenol	ND		5.0	1.0	ug/L		08/29/18 12:59	09/04/18 19:09	1
Dibenzofuran	ND		0.40	0.060	ug/L		08/29/18 12:59	09/04/18 19:09	1
4-Bromophenyl phenyl ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Diethyl phthalate	ND		12	2.8	ug/L		08/29/18 12:59	09/04/18 19:09	1
Dimethyl phthalate	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Anthracene	ND		15	0.020	ug/L		08/29/18 12:59	09/04/18 19:09	1
Di-n-butyl phthalate	ND		3.0	0.55	ug/L		08/29/18 12:59	09/04/18 19:09	1
Fluorene	ND		2.0	0.090	ug/L		08/29/18 12:59	09/04/18 19:09	1
Fluoranthene	ND		3.0	0.15	ug/L		08/29/18 12:59	09/04/18 19:09	1
Hexachlorobenzene	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Hexachlorobutadiene	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Butyl benzyl phthalate	ND		10	4.0	ug/L		08/29/18 12:59	09/04/18 19:09	1
Hexachlorocyclopentadiene	ND		3.0	1.3	ug/L		08/29/18 12:59	09/04/18 19:09	1
3,3'-Dichlorobenzidine	ND		15	1.1	ug/L		08/29/18 12:59	09/04/18 19:09	1
Hexachloroethane	ND		1.0	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzo[a]anthracene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 19:09	1
Chrysene	ND		0.60	0.17	ug/L		08/29/18 12:59	09/04/18 19:09	1
Isophorone	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Bis(2-ethylhexyl) phthalate	ND		15	6.3	ug/L		08/29/18 12:59	09/04/18 19:09	1
Naphthalene	ND		0.40	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Di-n-octyl phthalate	ND		1.0	0.18	ug/L		08/29/18 12:59	09/04/18 19:09	1
Nitrobenzene	ND		0.60	0.22	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzo[a]pyrene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 19:09	1
Indeno[1,2,3-cd]pyrene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 19:09	1
N-Nitrosodi-n-propylamine	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Dibenz(a,h)anthracene	ND		0.60	0.020	ug/L		08/29/18 12:59	09/04/18 19:09	1
N-Nitrosodiphenylamine	ND		3.0	0.60	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzo[g,h,i]perylene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 19:09	1
Pentachlorophenol	ND		10	2.5	ug/L		08/29/18 12:59	09/04/18 19:09	1
Carbazole	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1
Phenanthrene	ND		1.0	0.13	ug/L		08/29/18 12:59	09/04/18 19:09	1
1-Methylnaphthalene	ND		1.0	0.070	ug/L		08/29/18 12:59	09/04/18 19:09	1
Phenol	ND		4.0	0.92	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzo[b]fluoranthene	ND		1.0	0.050	ug/L		08/29/18 12:59	09/04/18 19:09	1
Benzo[k]fluoranthene	ND		1.0	0.020	ug/L		08/29/18 12:59	09/04/18 19:09	1
Pyrene	ND		2.0	0.39	ug/L		08/29/18 12:59	09/04/18 19:09	1
bis(chloroisopropyl) ether	ND		0.60	0.10	ug/L		08/29/18 12:59	09/04/18 19:09	1

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*67*		*48 - 125*	*08/29/18 12:59*	*09/04/18 19:09*	*1*
2-Fluorobiphenyl	*90*		*50 - 120*	*08/29/18 12:59*	*09/04/18 19:09*	*1*
2-Fluorophenol (Surr)	*76*		*50 - 120*	*08/29/18 12:59*	*09/04/18 19:09*	*1*
Nitrobenzene-d5 (Surr)	*95*		*62 - 120*	*08/29/18 12:59*	*09/04/18 19:09*	*1*
Phenol-d5 (Surr)	*75*		*52 - 120*	*08/29/18 12:59*	*09/04/18 19:09*	*1*
Terphenyl-d14 (Surr)	*103*		*55 - 126*	*08/29/18 12:59*	*09/04/18 19:09*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCS 580-282779/2-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 282779

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,3-Dichlorobenzene	2.00	1.43		ug/L		71	42 - 120
1,4-Dichlorobenzene	2.00	1.48		ug/L		74	40 - 120
1,2-Dichlorobenzene	2.00	1.60		ug/L		80	44 - 120
2-Chlorophenol	2.00	1.78		ug/L		89	60 - 125
2-Methylphenol	2.00	1.78		ug/L		89	53 - 120
2,4-Dimethylphenol	2.00	1.17	J	ug/L		58	20 - 120
2-Nitrophenol	2.00	1.88		ug/L		94	52 - 127
3 & 4 Methylphenol	2.00	1.84		ug/L		92	54 - 120
2,4-Dichlorophenol	2.00	1.79	J	ug/L		89	68 - 120
1,2,4-Trichlorobenzene	2.00	1.57		ug/L		79	46 - 120
4-Chloroaniline	2.00	ND	*	ug/L		17	20 - 129
4-Chloro-3-methylphenol	2.00	1.92		ug/L		96	52 - 126
2-Methylnaphthalene	2.00	1.65		ug/L		83	62 - 120
2,4,6-Trichlorophenol	2.00	1.73		ug/L		87	55 - 136
2,4,5-Trichlorophenol	2.00	1.67		ug/L		84	56 - 122
2-Chloronaphthalene	2.00	1.61		ug/L		80	53 - 120
2-Nitroaniline	2.00	1.86		ug/L		93	50 - 127
Acenaphthylene	2.00	1.72		ug/L		86	43 - 120
2,6-Dinitrotoluene	2.00	1.84		ug/L		92	68 - 123
3-Nitroaniline	2.00	1.16	J	ug/L		58	39 - 120
Acenaphthene	2.00	1.86		ug/L		93	62 - 120
Benzoic acid	4.00	1.36	J	ug/L		34	20 - 145
2,4-Dinitrophenol	4.00	2.24	J	ug/L		56	20 - 140
Benzyl alcohol	2.00	2.86	J *	ug/L		143	20 - 140
4-Nitrophenol	4.00	3.75	J	ug/L		94	40 - 150
Bis(2-chloroethoxy)methane	2.00	1.98		ug/L		99	59 - 120
Bis(2-chloroethyl)ether	2.00	1.97		ug/L		99	64 - 120
2,4-Dinitrotoluene	2.00	1.86		ug/L		93	71 - 123
4-Chlorophenyl phenyl ether	2.00	1.76		ug/L		88	61 - 125
4-Nitroaniline	2.00	1.50	J	ug/L		75	40 - 120
4,6-Dinitro-2-methylphenol	4.00	2.73	J	ug/L		68	20 - 144
Dibenzofuran	2.00	1.72		ug/L		86	60 - 125
4-Bromophenyl phenyl ether	2.00	1.86		ug/L		93	62 - 120
Diethyl phthalate	2.00	ND		ug/L		115	66 - 124
Dimethyl phthalate	2.00	2.06		ug/L		103	64 - 128
Anthracene	2.00	1.62	J	ug/L		81	34 - 125
Di-n-butyl phthalate	2.00	2.29	J	ug/L		115	57 - 146
Fluorene	2.00	1.74	J	ug/L		87	64 - 125
Fluoranthene	2.00	2.09	J	ug/L		105	70 - 120
Hexachlorobenzene	2.00	1.90		ug/L		95	55 - 120
Hexachlorobutadiene	2.00	1.27		ug/L		63	27 - 120
Butyl benzyl phthalate	2.00	ND		ug/L		135	64 - 150
Hexachlorocyclopentadiene	2.00	ND	*	ug/L		17	20 - 120
3,3'-Dichlorobenzidine	4.00	2.63	J	ug/L		66	20 - 146
Hexachloroethane	2.00	1.29		ug/L		64	30 - 120
Benzo[a]anthracene	2.00	2.06		ug/L		103	59 - 129
Chrysene	2.00	1.95		ug/L		98	65 - 120
Isophorone	2.00	2.05		ug/L		102	64 - 125

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCS 580-282779/2-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 282779

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Bis(2-ethylhexyl) phthalate	2.00	ND		ug/L		124	20 - 150
Naphthalene	2.00	1.66		ug/L		83	63 - 120
Di-n-octyl phthalate	2.00	2.18		ug/L		109	58 - 143
Nitrobenzene	2.00	2.02		ug/L		101	61 - 121
Benzo[a]pyrene	2.00	1.40		ug/L		70	28 - 133
Indeno[1,2,3-cd]pyrene	2.00	1.88		ug/L		94	55 - 142
N-Nitrosodi-n-propylamine	2.00	1.94		ug/L		97	54 - 120
Dibenz(a,h)anthracene	2.00	1.99		ug/L		100	56 - 146
N-Nitrosodiphenylamine	2.00	1.70	J	ug/L		85	33 - 120
Benzo[g,h,i]perylene	2.00	1.96		ug/L		98	56 - 146
Pentachlorophenol	4.00	ND		ug/L		60	20 - 120
Carbazole	2.00	2.22		ug/L		111	58 - 135
Phenanthrene	2.00	1.94		ug/L		97	63 - 120
1-Methylnaphthalene	2.00	1.75		ug/L		87	65 - 120
Phenol	2.00	1.94	J	ug/L		97	41 - 135
Benzo[b]fluoranthene	2.00	1.75		ug/L		88	62 - 141
Benzo[k]fluoranthene	2.00	1.79		ug/L		90	57 - 135
Pyrene	2.00	1.93	J	ug/L		96	64 - 120
bis(chloroisopropyl) ether	2.00	2.03		ug/L		101	32 - 133

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
2,4,6-Tribromophenol (Surr)	*97*		*48 - 125*
2-Fluorobiphenyl	*89*		*50 - 120*
2-Fluorophenol (Surr)	*86*		*50 - 120*
Nitrobenzene-d5 (Surr)	*105*		*62 - 120*
Phenol-d5 (Surr)	*87*		*52 - 120*
Terphenyl-d14 (Surr)	*108*		*55 - 126*

Lab Sample ID: LCSD 580-282779/3-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 282779

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,3-Dichlorobenzene	2.00	1.37		ug/L		69	42 - 120	4	35
1,4-Dichlorobenzene	2.00	1.46		ug/L		73	40 - 120	2	35
1,2-Dichlorobenzene	2.00	1.58		ug/L		79	44 - 120	1	32
2-Chlorophenol	2.00	1.81		ug/L		90	60 - 125	2	34
2-Methylphenol	2.00	1.86		ug/L		93	53 - 120	4	26
2,4-Dimethylphenol	2.00	1.24	J	ug/L		62	20 - 120	6	35
2-Nitrophenol	2.00	1.90		ug/L		95	52 - 127	1	25
3 & 4 Methylphenol	2.00	1.90		ug/L		95	54 - 120	4	27
2,4-Dichlorophenol	2.00	1.95	J	ug/L		98	68 - 120	9	20
1,2,4-Trichlorobenzene	2.00	1.54		ug/L		77	46 - 120	2	32
4-Chloroaniline	2.00	ND	*	ug/L		25	20 - 129	36	35
4-Chloro-3-methylphenol	2.00	2.05		ug/L		102	52 - 126	6	26
2-Methylnaphthalene	2.00	1.74		ug/L		87	62 - 120	5	20
2,4,6-Trichlorophenol	2.00	1.95		ug/L		98	55 - 136	12	20
2,4,5-Trichlorophenol	2.00	1.83		ug/L		91	56 - 122	9	25

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCSD 580-282779/3-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 282779

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
2-Chloronaphthalene	2.00	1.79		ug/L		89	53 - 120	11	20
2-Nitroaniline	2.00	2.00		ug/L		100	50 - 127	7	29
Acenaphthylene	2.00	1.93		ug/L		96	43 - 120	11	20
2,6-Dinitrotoluene	2.00	1.90		ug/L		95	68 - 123	3	14
3-Nitroaniline	2.00	1.24	J	ug/L		62	39 - 120	7	23
Acenaphthene	2.00	2.05		ug/L		102	62 - 120	10	26
Benzoic acid	4.00	1.23	J	ug/L		31	20 - 145	10	35
2,4-Dinitrophenol	4.00	2.51	J	ug/L		63	20 - 140	11	35
Benzyl alcohol	2.00	2.65	J	ug/L		132	20 - 140	8	35
4-Nitrophenol	4.00	4.25	J	ug/L		106	40 - 150	13	35
Bis(2-chloroethoxy)methane	2.00	1.97		ug/L		99	59 - 120	0	19
Bis(2-chloroethyl)ether	2.00	1.98		ug/L		99	64 - 120	0	20
2,4-Dinitrotoluene	2.00	2.02		ug/L		101	71 - 123	8	15
4-Chlorophenyl phenyl ether	2.00	1.91		ug/L		95	61 - 125	8	14
4-Nitroaniline	2.00	1.61	J	ug/L		81	40 - 120	7	18
4,6-Dinitro-2-methylphenol	4.00	2.81	J	ug/L		70	20 - 144	3	35
Dibenzofuran	2.00	1.91		ug/L		95	60 - 125	10	20
4-Bromophenyl phenyl ether	2.00	1.84		ug/L		92	62 - 120	1	20
Diethyl phthalate	2.00	ND		ug/L		118	66 - 124	3	35
Dimethyl phthalate	2.00	2.20		ug/L		110	64 - 128	6	14
Anthracene	2.00	1.62	J	ug/L		81	34 - 125	0	26
Di-n-butyl phthalate	2.00	2.19	J	ug/L		110	57 - 146	4	20
Fluorene	2.00	1.94	J	ug/L		97	64 - 125	11	20
Fluoranthene	2.00	2.08	J	ug/L		104	70 - 120	0	20
Hexachlorobenzene	2.00	1.91		ug/L		96	55 - 120	1	20
Hexachlorobutadiene	2.00	1.22		ug/L		61	27 - 120	4	31
Butyl benzyl phthalate	2.00	ND		ug/L		126	64 - 150	7	20
Hexachlorocyclopentadiene	2.00	ND	*	ug/L		15	20 - 120	13	35
3,3'-Dichlorobenzidine	4.00	3.46	J	ug/L		87	20 - 146	27	35
Hexachloroethane	2.00	1.16		ug/L		58	30 - 120	10	35
Benzo[a]anthracene	2.00	2.07		ug/L		104	59 - 129	1	27
Chrysene	2.00	1.98		ug/L		99	65 - 120	1	26
Isophorone	2.00	2.07		ug/L		103	64 - 125	1	20
Bis(2-ethylhexyl) phthalate	2.00	ND	*	ug/L		162	20 - 150	27	35
Naphthalene	2.00	1.71		ug/L		85	63 - 120	3	20
Di-n-octyl phthalate	2.00	2.24		ug/L		112	58 - 143	3	20
Nitrobenzene	2.00	2.06		ug/L		103	61 - 121	2	20
Benzo[a]pyrene	2.00	1.51		ug/L		76	28 - 133	8	29
Indeno[1,2,3-cd]pyrene	2.00	1.98		ug/L		99	55 - 142	5	20
N-Nitrosodi-n-propylamine	2.00	2.03		ug/L		102	54 - 120	4	20
Dibenz(a,h)anthracene	2.00	2.07		ug/L		104	56 - 146	4	28
N-Nitrosodiphenylamine	2.00	1.70	J	ug/L		85	33 - 120	0	35
Benzo[g,h,i]perylene	2.00	2.01		ug/L		100	56 - 146	3	20
Pentachlorophenol	4.00	ND		ug/L		56	20 - 120	7	35
Carbazole	2.00	2.22		ug/L		111	58 - 135	0	20
Phenanthrene	2.00	1.93		ug/L		97	63 - 120	0	20
1-Methylnaphthalene	2.00	1.77		ug/L		88	65 - 120	1	20
Phenol	2.00	1.97	J	ug/L		98	41 - 135	1	34

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCSD 580-282779/3-A
Matrix: Water
Analysis Batch: 283155

Client Sample ID: Lab Control Sample Dup
Prep Type: Total/NA
Prep Batch: 282779

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Benzo[b]fluoranthene			2.00	1.84		ug/L		92	62 - 141	5	20
Benzo[k]fluoranthene			2.00	1.90		ug/L		95	57 - 135	6	20
Pyrene			2.00	1.92	J	ug/L		96	64 - 120	1	20
bis(chloroisopropyl) ether			2.00	2.09		ug/L		104	32 - 133	3	20

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
2,4,6-Tribromophenol (Surr)	*91*		*48 - 125*
2-Fluorobiphenyl	*96*		*50 - 120*
2-Fluorophenol (Surr)	*89*		*50 - 120*
Nitrobenzene-d5 (Surr)	*107*		*62 - 120*
Phenol-d5 (Surr)	*87*		*52 - 120*
Terphenyl-d14 (Surr)	*107*		*55 - 126*

Lab Sample ID: MB 580-283435/1-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Method Blank
Prep Type: SPLP West
Prep Batch: 283880

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,3-Dichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
1,4-Dichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
1,2-Dichlorobenzene	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Chlorophenol	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Methylphenol	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4-Dimethylphenol	ND		20	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Nitrophenol	ND		5.0	0.75	ug/L		09/13/18 10:05	09/13/18 20:23	1
3 & 4 Methylphenol	ND		4.0	0.15	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4-Dichlorophenol	ND		20	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
1,2,4-Trichlorobenzene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Chloroaniline	ND		50	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Chloro-3-methylphenol	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Methylnaphthalene	ND		2.0	0.15	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4,6-Trichlorophenol	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4,5-Trichlorophenol	ND		2.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Chloronaphthalene	ND		5.0	0.15	ug/L		09/13/18 10:05	09/13/18 20:23	1
2-Nitroaniline	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
Acenaphthylene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,6-Dinitrotoluene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
3-Nitroaniline	ND		15	0.15	ug/L		09/13/18 10:05	09/13/18 20:23	1
Acenaphthene	ND		2.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzoic acid	ND		20	3.5	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4-Dinitrophenol	ND		25	1.4	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzyl alcohol	ND		15	0.75	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Nitrophenol	ND		75	2.6	ug/L		09/13/18 10:05	09/13/18 20:23	1
Bis(2-chloroethoxy)methane	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Bis(2-chloroethyl)ether	ND		3.0	0.15	ug/L		09/13/18 10:05	09/13/18 20:23	1
2,4-Dinitrotoluene	ND		5.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Chlorophenyl phenyl ether	ND		3.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Nitroaniline	ND		10	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: MB 580-283435/1-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Method Blank
Prep Type: SPLP West
Prep Batch: 283880

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
4,6-Dinitro-2-methylphenol	ND		25	1.3	ug/L		09/13/18 10:05	09/13/18 20:23	1
Dibenzofuran	ND		2.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
4-Bromophenyl phenyl ether	ND		3.0	0.10	ug/L		09/13/18 10:05	09/13/18 20:23	1
Diethyl phthalate	ND		60	2.8	ug/L		09/13/18 10:05	09/13/18 20:23	1
Dimethyl phthalate	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
Anthracene	ND		75	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Di-n-butyl phthalate	ND		15	2.2	ug/L		09/13/18 10:05	09/13/18 20:23	1
Fluorene	ND		10	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Fluoranthene	ND		15	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
Hexachlorobenzene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Hexachlorobutadiene	ND		5.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
Butyl benzyl phthalate	ND		50	4.7	ug/L		09/13/18 10:05	09/13/18 20:23	1
Hexachlorocyclopentadiene	ND		25	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
3,3'-Dichlorobenzidine	ND		75	0.95	ug/L		09/13/18 10:05	09/13/18 20:23	1
Hexachloroethane	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzo[a]anthracene	ND		5.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Chrysene	ND		3.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Isophorone	ND		2.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
Bis(2-ethylhexyl) phthalate	ND		75	13	ug/L		09/13/18 10:05	09/13/18 20:23	1
Naphthalene	ND		2.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Di-n-octyl phthalate	ND	*	5.0	0.65	ug/L		09/13/18 10:05	09/13/18 20:23	1
Nitrobenzene	ND		5.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzo[a]pyrene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Indeno[1,2,3-cd]pyrene	ND	*	5.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
N-Nitrosodi-n-propylamine	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1
Dibenz(a,h)anthracene	ND	*	3.0	0.35	ug/L		09/13/18 10:05	09/13/18 20:23	1
N-Nitrosodiphenylamine	ND		75	16	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzo[g,h,i]perylene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Pentachlorophenol	ND		50	1.3	ug/L		09/13/18 10:05	09/13/18 20:23	1
Carbazole	ND		3.0	0.50	ug/L		09/13/18 10:05	09/13/18 20:23	1
Phenanthrene	ND		5.0	1.2	ug/L		09/13/18 10:05	09/13/18 20:23	1
1-Methylnaphthalene	ND		5.0	0.10	ug/L		09/13/18 10:05	09/13/18 20:23	1
Phenol	ND		20	1.8	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzo[b]fluoranthene	ND	*	5.0	0.20	ug/L		09/13/18 10:05	09/13/18 20:23	1
Benzo[k]fluoranthene	ND	*	5.0	0.25	ug/L		09/13/18 10:05	09/13/18 20:23	1
Pyrene	ND		10	0.55	ug/L		09/13/18 10:05	09/13/18 20:23	1
bis(chloroisopropyl) ether	ND		3.0	0.30	ug/L		09/13/18 10:05	09/13/18 20:23	1

Surrogate	*MB %Recovery*	*MB Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
2,4,6-Tribromophenol (Surr)	*87*		*28 - 131*	*09/13/18 10:05*	*09/13/18 20:23*	*1*
2-Fluorobiphenyl	*81*		*63 - 120*	*09/13/18 10:05*	*09/13/18 20:23*	*1*
2-Fluorophenol (Surr)	*84*		*20 - 147*	*09/13/18 10:05*	*09/13/18 20:23*	*1*
Nitrobenzene-d5 (Surr)	*93*		*60 - 120*	*09/13/18 10:05*	*09/13/18 20:23*	*1*
Phenol-d5 (Surr)	*61*		*21 - 135*	*09/13/18 10:05*	*09/13/18 20:23*	*1*
Terphenyl-d14 (Surr)	*100*		*66 - 120*	*09/13/18 10:05*	*09/13/18 20:23*	*1*

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCS 580-283435/2-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Lab Control Sample
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
1,3-Dichlorobenzene	10.0	5.70	*	ug/L		57	75 - 120
1,4-Dichlorobenzene	10.0	7.34		ug/L		73	46 - 120
1,2-Dichlorobenzene	10.0	7.01		ug/L		70	47 - 120
2-Chlorophenol	10.0	5.78		ug/L		58	56 - 124
2-Methylphenol	10.0	7.31		ug/L		73	49 - 120
2,4-Dimethylphenol	10.0	7.49	J	ug/L		75	20 - 120
2-Nitrophenol	10.0	8.66		ug/L		87	20 - 150
3 & 4 Methylphenol	10.0	7.50		ug/L		75	50 - 124
2,4-Dichlorophenol	10.0	7.10	J *	ug/L		71	72 - 120
1,2,4-Trichlorobenzene	10.0	6.43		ug/L		64	53 - 120
4-Chloroaniline	10.0	6.57	J	ug/L		66	20 - 131
4-Chloro-3-methylphenol	10.0	8.70		ug/L		87	62 - 120
2-Methylnaphthalene	10.0	7.36		ug/L		74	63 - 120
2,4,6-Trichlorophenol	10.0	8.57		ug/L		86	64 - 121
2,4,5-Trichlorophenol	10.0	8.93		ug/L		89	67 - 120
2-Chloronaphthalene	10.0	7.93		ug/L		79	60 - 120
2-Nitroaniline	10.0	8.65		ug/L		87	54 - 150
Acenaphthylene	10.0	8.23		ug/L		82	31 - 135
2,6-Dinitrotoluene	10.0	11.5		ug/L		115	65 - 136
3-Nitroaniline	10.0	8.83	J	ug/L		88	20 - 130
Acenaphthene	10.0	8.37		ug/L		84	59 - 120
Benzoic acid	20.0	17.7	J	ug/L		89	20 - 150
2,4-Dinitrophenol	20.0	16.5	J	ug/L		82	20 - 150
Benzyl alcohol	10.0	6.64	J	ug/L		66	41 - 139
4-Nitrophenol	20.0	16.9	J	ug/L		85	20 - 150
Bis(2-chloroethoxy)methane	10.0	6.86		ug/L		69	66 - 122
Bis(2-chloroethyl)ether	10.0	4.80	*	ug/L		48	66 - 120
2,4-Dinitrotoluene	10.0	9.66		ug/L		97	63 - 133
4-Chlorophenyl phenyl ether	10.0	8.06		ug/L		81	63 - 120
4-Nitroaniline	10.0	10.3		ug/L		103	40 - 121
4,6-Dinitro-2-methylphenol	20.0	19.1	J	ug/L		95	20 - 150
Dibenzofuran	10.0	7.73		ug/L		77	64 - 120
4-Bromophenyl phenyl ether	10.0	8.52		ug/L		85	67 - 120
Diethyl phthalate	10.0	10.2	J	ug/L		102	55 - 145
Dimethyl phthalate	10.0	8.73		ug/L		87	68 - 125
Anthracene	10.0	8.63	J	ug/L		86	20 - 139
Di-n-butyl phthalate	10.0	9.75	J	ug/L		97	71 - 150
Fluorene	10.0	8.21	J	ug/L		82	65 - 126
Fluoranthene	10.0	7.65	J	ug/L		76	68 - 130
Hexachlorobenzene	10.0	9.76		ug/L		98	60 - 120
Hexachlorobutadiene	10.0	7.34		ug/L		73	31 - 120
Butyl benzyl phthalate	10.0	15.7	J *	ug/L		157	72 - 150
Hexachlorocyclopentadiene	10.0	7.54	J	ug/L		75	20 - 120
3,3'-Dichlorobenzidine	20.0	18.2	J	ug/L		91	20 - 150
Hexachloroethane	10.0	8.20		ug/L		82	38 - 120
Benzo[a]anthracene	10.0	9.41		ug/L		94	60 - 124
Chrysene	10.0	7.91		ug/L		79	68 - 120
Isophorone	10.0	7.01		ug/L		70	68 - 128

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCS 580-283435/2-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Lab Control Sample
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Bis(2-ethylhexyl) phthalate	10.0	ND		ug/L		99	37 - 150
Naphthalene	10.0	8.12		ug/L		81	64 - 120
Di-n-octyl phthalate	10.0	10.9		ug/L		109	68 - 150
Nitrobenzene	10.0	7.29		ug/L		73	60 - 130
Benzo[a]pyrene	10.0	8.37		ug/L		84	20 - 132
Indeno[1,2,3-cd]pyrene	10.0	7.49		ug/L		75	68 - 127
N-Nitrosodi-n-propylamine	10.0	6.71		ug/L		67	61 - 120
Dibenz(a,h)anthracene	10.0	7.93		ug/L		79	56 - 130
N-Nitrosodiphenylamine	10.0	ND		ug/L		82	36 - 120
Benzo[g,h,i]perylene	10.0	6.84		ug/L		68	59 - 131
Pentachlorophenol	20.0	15.9	J	ug/L		80	39 - 147
Carbazole	10.0	10.1		ug/L		101	78 - 126
Phenanthrene	10.0	7.99		ug/L		80	64 - 120
1-Methylnaphthalene	10.0	7.61		ug/L		76	63 - 120
Phenol	10.0	3.96	J	ug/L		40	30 - 140
Benzo[b]fluoranthene	10.0	8.66		ug/L		87	69 - 123
Benzo[k]fluoranthene	10.0	8.36		ug/L		84	69 - 121
Pyrene	10.0	8.01	J	ug/L		80	63 - 126
bis(chloroisopropyl) ether	10.0	7.44		ug/L		74	65 - 121

Surrogate	*LCS %Recovery*	*LCS Qualifier*	*Limits*
2,4,6-Tribromophenol (Surr)	*82*		*28 - 131*
2-Fluorobiphenyl	*78*		*63 - 120*
2-Fluorophenol (Surr)	*45*		*20 - 147*
Nitrobenzene-d5 (Surr)	*69*		*60 - 120*
Phenol-d5 (Surr)	*36*		*21 - 135*
Terphenyl-d14 (Surr)	*85*		*66 - 120*

Lab Sample ID: LCSD 580-283435/3-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
1,3-Dichlorobenzene	10.0	6.74	*	ug/L		67	75 - 120	17	33
1,4-Dichlorobenzene	10.0	6.90		ug/L		69	46 - 120	6	30
1,2-Dichlorobenzene	10.0	7.50		ug/L		75	47 - 120	7	29
2-Chlorophenol	10.0	8.31	*	ug/L		83	56 - 124	36	35
2-Methylphenol	10.0	8.97		ug/L		90	49 - 120	20	35
2,4-Dimethylphenol	10.0	9.32	J	ug/L		93	20 - 120	22	35
2-Nitrophenol	10.0	11.0		ug/L		110	20 - 150	24	30
3 & 4 Methylphenol	10.0	8.53		ug/L		85	50 - 124	13	35
2,4-Dichlorophenol	10.0	8.92	J	ug/L		89	72 - 120	23	35
1,2,4-Trichlorobenzene	10.0	8.93	*	ug/L		89	53 - 120	33	25
4-Chloroaniline	10.0	7.49	J	ug/L		75	20 - 131	13	35
4-Chloro-3-methylphenol	10.0	8.73		ug/L		87	62 - 120	0	23
2-Methylnaphthalene	10.0	7.19		ug/L		72	63 - 120	2	19
2,4,6-Trichlorophenol	10.0	9.58		ug/L		96	64 - 121	11	26
2,4,5-Trichlorophenol	10.0	8.76		ug/L		88	67 - 120	2	26

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCSD 580-283435/3-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
2-Chloronaphthalene	10.0	7.87		ug/L		79	60 - 120	1	20
2-Nitroaniline	10.0	9.14		ug/L		91	54 - 150	5	20
Acenaphthylene	10.0	8.34		ug/L		83	31 - 135	1	18
2,6-Dinitrotoluene	10.0	11.4		ug/L		114	65 - 136	1	21
3-Nitroaniline	10.0	10.2	J	ug/L		102	20 - 130	14	35
Acenaphthene	10.0	9.83	*	ug/L		98	59 - 120	16	15
Benzoic acid	20.0	20.6		ug/L		103	20 - 150	15	35
2,4-Dinitrophenol	20.0	16.6	J	ug/L		83	20 - 150	1	35
Benzyl alcohol	10.0	7.81	J	ug/L		78	41 - 139	16	35
4-Nitrophenol	20.0	17.8	J	ug/L		89	20 - 150	5	35
Bis(2-chloroethoxy)methane	10.0	9.20	*	ug/L		92	66 - 122	29	21
Bis(2-chloroethyl)ether	10.0	8.08	*	ug/L		81	66 - 120	51	23
2,4-Dinitrotoluene	10.0	10.5		ug/L		105	63 - 133	8	23
4-Chlorophenyl phenyl ether	10.0	9.06		ug/L		91	63 - 120	12	19
4-Nitroaniline	10.0	8.87	J	ug/L		89	40 - 121	15	33
4,6-Dinitro-2-methylphenol	20.0	20.1	J	ug/L		101	20 - 150	5	35
Dibenzofuran	10.0	8.48		ug/L		85	64 - 120	9	17
4-Bromophenyl phenyl ether	10.0	10.6		ug/L		106	67 - 120	22	23
Diethyl phthalate	10.0	11.1	J	ug/L		111	55 - 145	8	35
Dimethyl phthalate	10.0	8.94		ug/L		89	68 - 125	2	19
Anthracene	10.0	9.67	J	ug/L		97	20 - 139	11	27
Di-n-butyl phthalate	10.0	12.3	J	ug/L		123	71 - 150	23	35
Fluorene	10.0	9.18	J	ug/L		92	65 - 126	11	17
Fluoranthene	10.0	9.46	J *	ug/L		95	68 - 130	21	20
Hexachlorobenzene	10.0	10.6		ug/L		106	60 - 120	8	23
Hexachlorobutadiene	10.0	8.45		ug/L		85	31 - 120	14	28
Butyl benzyl phthalate	10.0	13.4	J	ug/L		134	72 - 150	16	35
Hexachlorocyclopentadiene	10.0	8.09	J	ug/L		81	20 - 120	7	35
3,3'-Dichlorobenzidine	20.0	21.1	J	ug/L		106	20 - 150	15	35
Hexachloroethane	10.0	9.15		ug/L		91	38 - 120	11	35
Benzo[a]anthracene	10.0	9.60		ug/L		96	60 - 124	2	17
Chrysene	10.0	9.01		ug/L		90	68 - 120	13	19
Isophorone	10.0	9.30	*	ug/L		93	68 - 128	28	19
Bis(2-ethylhexyl) phthalate	10.0	ND		ug/L		101	37 - 150	2	35
Naphthalene	10.0	9.95		ug/L		99	64 - 120	20	20
Di-n-octyl phthalate	10.0	11.8		ug/L		118	68 - 150	7	21
Nitrobenzene	10.0	8.64		ug/L		86	60 - 130	17	31
Benzo[a]pyrene	10.0	8.45		ug/L		85	20 - 132	1	35
Indeno[1,2,3-cd]pyrene	10.0	9.56		ug/L		96	68 - 127	24	32
N-Nitrosodi-n-propylamine	10.0	8.36		ug/L		84	61 - 120	22	24
Dibenz(a,h)anthracene	10.0	9.94	*	ug/L		99	56 - 130	23	22
N-Nitrosodiphenylamine	10.0	ND		ug/L		97	36 - 120	17	21
Benzo[g,h,i]perylene	10.0	9.79	*	ug/L		98	59 - 131	36	25
Pentachlorophenol	20.0	15.9	J	ug/L		79	39 - 147	0	23
Carbazole	10.0	11.6		ug/L		116	78 - 126	14	20
Phenanthrene	10.0	9.03		ug/L		90	64 - 120	12	19
1-Methylnaphthalene	10.0	8.15		ug/L		82	63 - 120	7	20
Phenol	10.0	5.61	J *	ug/L		56	30 - 140	34	30

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: LCSD 580-283435/3-B
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: Lab Control Sample Dup
Prep Type: SPLP West
Prep Batch: 283880

Analyte			Spike Added	LCSD Result	LCSD Qualifier	Unit	D	%Rec	%Rec. Limits	RPD	RPD Limit
Benzo[b]fluoranthene			10.0	9.46		ug/L		95	69 - 123	9	20
Benzo[k]fluoranthene			10.0	8.84		ug/L		88	69 - 121	6	27
Pyrene			10.0	9.94	J *	ug/L		99	63 - 126	22	19
bis(chloroisopropyl) ether			10.0	8.79		ug/L		88	65 - 121	17	20

Surrogate	*LCSD %Recovery*	*LCSD Qualifier*	*Limits*
2,4,6-Tribromophenol (Surr)	*107*		*28 - 131*
2-Fluorobiphenyl	*77*		*63 - 120*
2-Fluorophenol (Surr)	*59*		*20 - 147*
Nitrobenzene-d5 (Surr)	*73*		*60 - 120*
Phenol-d5 (Surr)	*44*		*21 - 135*
Terphenyl-d14 (Surr)	*95*		*66 - 120*

Lab Sample ID: 580-79910-1 MS
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: SITK-18-AS-1
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec. Limits
1,3-Dichlorobenzene	ND	*	10.0	7.46		ug/L		75	75 - 120
1,4-Dichlorobenzene	ND		10.0	7.80		ug/L		78	46 - 120
1,2-Dichlorobenzene	ND		10.0	7.36		ug/L		74	47 - 120
2-Chlorophenol	ND	*	10.0	8.62		ug/L		86	56 - 124
2-Methylphenol	ND		10.0	7.55		ug/L		75	49 - 120
2,4-Dimethylphenol	ND		10.0	ND		ug/L		85	20 - 120
2-Nitrophenol	ND		10.0	11.0		ug/L		110	20 - 150
3 & 4 Methylphenol	ND		10.0	8.58		ug/L		86	50 - 124
2,4-Dichlorophenol	ND	*	10.0	ND		ug/L		80	72 - 120
1,2,4-Trichlorobenzene	ND	*	10.0	7.31		ug/L		73	53 - 120
4-Chloroaniline	ND		10.0	ND		ug/L		55	20 - 131
4-Chloro-3-methylphenol	ND		10.0	7.61		ug/L		76	62 - 120
2-Methylnaphthalene	ND		10.0	8.57		ug/L		84	63 - 120
2,4,6-Trichlorophenol	ND		10.0	8.06		ug/L		81	64 - 121
2,4,5-Trichlorophenol	ND		10.0	8.04		ug/L		80	67 - 120
2-Chloronaphthalene	ND	F1	10.0	5.94	F1	ug/L		59	60 - 120
2-Nitroaniline	ND		10.0	8.69		ug/L		87	54 - 150
Acenaphthylene	ND		10.0	6.49		ug/L		65	31 - 135
2,6-Dinitrotoluene	ND		10.0	8.67		ug/L		87	65 - 136
3-Nitroaniline	ND		10.0	ND		ug/L		73	20 - 130
Acenaphthene	ND	*	10.0	7.27		ug/L		67	59 - 120
Benzoic acid	ND		20.0	30.0		ug/L		105	20 - 150
2,4-Dinitrophenol	ND		20.0	ND		ug/L		84	20 - 150
Benzyl alcohol	ND		10.0	ND		ug/L		80	41 - 139
4-Nitrophenol	ND	F1	20.0	ND	F1	ug/L		194	20 - 150
Bis(2-chloroethoxy)methane	ND	*	10.0	7.19		ug/L		72	66 - 122
Bis(2-chloroethyl)ether	ND	*	10.0	8.02		ug/L		80	66 - 120
2,4-Dinitrotoluene	ND		10.0	10.1		ug/L		101	63 - 133
4-Chlorophenyl phenyl ether	ND		10.0	7.07		ug/L		71	63 - 120
4-Nitroaniline	ND		10.0	ND		ug/L		63	40 - 121

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Continued)

Lab Sample ID: 580-79910-1 MS
Matrix: Solid
Analysis Batch: 283944

Client Sample ID: SITK-18-AS-1
Prep Type: SPLP West
Prep Batch: 283880

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec. Limits
4,6-Dinitro-2-methylphenol	ND		20.0	ND		ug/L		93	20 - 150
Dibenzofuran	ND		10.0	7.06		ug/L		68	64 - 120
4-Bromophenyl phenyl ether	ND		10.0	7.30		ug/L		73	67 - 120
Diethyl phthalate	ND		10.0	ND		ug/L		88	55 - 145
Dimethyl phthalate	ND	F1	10.0	6.67	F1	ug/L		67	68 - 125
Anthracene	ND		10.0	ND		ug/L		80	20 - 139
Di-n-butyl phthalate	ND		10.0	ND		ug/L		96	71 - 150
Fluorene	ND		10.0	ND		ug/L		66	65 - 126
Fluoranthene	ND	*	10.0	ND		ug/L		76	68 - 130
Hexachlorobenzene	ND		10.0	8.11		ug/L		81	60 - 120
Hexachlorobutadiene	ND		10.0	8.56		ug/L		86	31 - 120
Butyl benzyl phthalate	ND	*	10.0	ND		ug/L		140	72 - 150
Hexachlorocyclopentadiene	ND		10.0	ND		ug/L		68	20 - 120
3,3'-Dichlorobenzidine	ND		20.0	ND		ug/L		42	20 - 150
Hexachloroethane	ND		10.0	8.15		ug/L		81	38 - 120
Benzo[a]anthracene	ND		10.0	10.4		ug/L		104	60 - 124
Chrysene	ND		10.0	8.68		ug/L		87	68 - 120
Isophorone	ND	*	10.0	7.26		ug/L		73	68 - 128
Bis(2-ethylhexyl) phthalate	ND		10.0	ND		ug/L		NC	37 - 150
Naphthalene	ND		10.0	8.86		ug/L		89	64 - 120
Di-n-octyl phthalate	ND		10.0	13.2		ug/L		132	68 - 150
Nitrobenzene	ND		10.0	8.88		ug/L		89	60 - 130
Benzo[a]pyrene	ND		10.0	8.35		ug/L		83	20 - 132
Indeno[1,2,3-cd]pyrene	ND		10.0	7.39		ug/L		74	68 - 127
N-Nitrosodi-n-propylamine	ND		10.0	7.65		ug/L		77	61 - 120
Dibenz(a,h)anthracene	ND	*	10.0	7.81		ug/L		78	56 - 130
N-Nitrosodiphenylamine	ND		10.0	ND		ug/L		NC	36 - 120
Benzo[g,h,i]perylene	ND	*	10.0	7.37		ug/L		74	59 - 131
Pentachlorophenol	ND		20.0	ND		ug/L		102	39 - 147
Carbazole	ND		10.0	9.24		ug/L		92	78 - 126
Phenanthrene	ND		10.0	8.01		ug/L		80	64 - 120
1-Methylnaphthalene	ND		10.0	10.5		ug/L		82	63 - 120
Phenol	ND	*	10.0	ND		ug/L		58	30 - 140
Benzo[b]fluoranthene	ND		10.0	8.18		ug/L		82	69 - 123
Benzo[k]fluoranthene	ND		10.0	8.54		ug/L		85	69 - 121
Pyrene	ND	*	10.0	ND		ug/L		77	63 - 126
bis(chloroisopropyl) ether	ND		10.0	8.14		ug/L		81	65 - 121

Surrogate	*MS %Recovery*	*MS Qualifier*	*Limits*
2,4,6-Tribromophenol (Surr)	*90*		*28 - 131*
2-Fluorobiphenyl	*59*	*X*	*63 - 120*
2-Fluorophenol (Surr)	*72*		*20 - 147*
Nitrobenzene-d5 (Surr)	*83*		*60 - 120*
Phenol-d5 (Surr)	*48*		*21 - 135*
Terphenyl-d14 (Surr)	*81*		*66 - 120*

GC/MS VOA

Analysis Batch: 283011

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-5	SITK-18-PW-1	Total/NA	Water	8260C	
580-79910-6	SITK-18-PW-4	Total/NA	Water	8260C	
580-79910-7	SITK-18-PW-2	Total/NA	Water	8260C	
580-79910-8	SITK-18-PW-3	Total/NA	Water	8260C	
580-79910-9	SITK-18-TB-PW-1	Total/NA	Water	8260C	
MB 580-283011/5	Method Blank	Total/NA	Water	8260C	
LCS 580-283011/6	Lab Control Sample	Total/NA	Water	8260C	
LCSD 580-283011/7	Lab Control Sample Dup	Total/NA	Water	8260C	

Prep Batch: 283200

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-2	SITK-18-TB-AS-1	Total/NA	Solid	5035	
MB 580-283200/1-A	Method Blank	Total/NA	Solid	5035	
LCS 580-283200/2-A	Lab Control Sample	Total/NA	Solid	5035	
LCSD 580-283200/3-A	Lab Control Sample Dup	Total/NA	Solid	5035	

Analysis Batch: 283203

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-2	SITK-18-TB-AS-1	Total/NA	Solid	8260C	283200
MB 580-283200/1-A	Method Blank	Total/NA	Solid	8260C	283200
LCS 580-283200/2-A	Lab Control Sample	Total/NA	Solid	8260C	283200
LCSD 580-283200/3-A	Lab Control Sample Dup	Total/NA	Solid	8260C	283200

Leach Batch: 283436

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP	Solid	1312	
580-79910-3	SITK-18-AS-2	SPLP	Solid	1312	
580-79910-4	SITK-18-AS-3	SPLP	Solid	1312	
MB 580-283436/1-A	Method Blank	SPLP	Solid	1312	
LCS 580-283436/2-A	Lab Control Sample	SPLP	Solid	1312	
LCSD 580-283436/3-A	Lab Control Sample Dup	SPLP	Solid	1312	

Analysis Batch: 283529

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP	Solid	8260C	283436
580-79910-3	SITK-18-AS-2	SPLP	Solid	8260C	283436
580-79910-4	SITK-18-AS-3	SPLP	Solid	8260C	283436
MB 580-283436/1-A	Method Blank	SPLP	Solid	8260C	283436
LCS 580-283436/2-A	Lab Control Sample	SPLP	Solid	8260C	283436
LCSD 580-283436/3-A	Lab Control Sample Dup	SPLP	Solid	8260C	283436

Analysis Batch: 284209

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP	Solid	8260C	283436
580-79910-3	SITK-18-AS-2	SPLP	Solid	8260C	283436
580-79910-4	SITK-18-AS-3	SPLP	Solid	8260C	283436
MB 580-283436/1-A	Method Blank	SPLP	Solid	8260C	283436
LCS 580-283436/2-A	Lab Control Sample	SPLP	Solid	8260C	283436
LCSD 580-283436/3-A	Lab Control Sample Dup	SPLP	Solid	8260C	283436

GC/MS Semi VOA

Prep Batch: 282779

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-5	SITK-18-PW-1	Total/NA	Water	3520C	
580-79910-6	SITK-18-PW-4	Total/NA	Water	3520C	
580-79910-7	SITK-18-PW-2	Total/NA	Water	3520C	
580-79910-8	SITK-18-PW-3	Total/NA	Water	3520C	
MB 580-282779/1-A	Method Blank	Total/NA	Water	3520C	
LCS 580-282779/2-A	Lab Control Sample	Total/NA	Water	3520C	
LCSD 580-282779/3-A	Lab Control Sample Dup	Total/NA	Water	3520C	

Analysis Batch: 283155

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-5	SITK-18-PW-1	Total/NA	Water	8270D	282779
580-79910-6	SITK-18-PW-4	Total/NA	Water	8270D	282779
580-79910-7	SITK-18-PW-2	Total/NA	Water	8270D	282779
580-79910-8	SITK-18-PW-3	Total/NA	Water	8270D	282779
MB 580-282779/1-A	Method Blank	Total/NA	Water	8270D	282779
LCS 580-282779/2-A	Lab Control Sample	Total/NA	Water	8270D	282779
LCSD 580-282779/3-A	Lab Control Sample Dup	Total/NA	Water	8270D	282779

Leach Batch: 283435

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP West	Solid	1312	
580-79910-3	SITK-18-AS-2	SPLP West	Solid	1312	
580-79910-4	SITK-18-AS-3	SPLP West	Solid	1312	
MB 580-283435/1-B	Method Blank	SPLP West	Solid	1312	
LCS 580-283435/2-B	Lab Control Sample	SPLP West	Solid	1312	
LCSD 580-283435/3-B	Lab Control Sample Dup	SPLP West	Solid	1312	
580-79910-1 MS	SITK-18-AS-1	SPLP West	Solid	1312	

Prep Batch: 283880

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP West	Solid	3510C	283435
580-79910-3	SITK-18-AS-2	SPLP West	Solid	3510C	283435
580-79910-4	SITK-18-AS-3	SPLP West	Solid	3510C	283435
MB 580-283435/1-B	Method Blank	SPLP West	Solid	3510C	283435
LCS 580-283435/2-B	Lab Control Sample	SPLP West	Solid	3510C	283435
LCSD 580-283435/3-B	Lab Control Sample Dup	SPLP West	Solid	3510C	283435
580-79910-1 MS	SITK-18-AS-1	SPLP West	Solid	3510C	283435

Analysis Batch: 283944

Lab Sample ID	Client Sample ID	Prep Type	Matrix	Method	Prep Batch
580-79910-1	SITK-18-AS-1	SPLP West	Solid	8270D	283880
580-79910-3	SITK-18-AS-2	SPLP West	Solid	8270D	283880
580-79910-4	SITK-18-AS-3	SPLP West	Solid	8270D	283880
MB 580-283435/1-B	Method Blank	SPLP West	Solid	8270D	283880
LCS 580-283435/2-B	Lab Control Sample	SPLP West	Solid	8270D	283880
LCSD 580-283435/3-B	Lab Control Sample Dup	SPLP West	Solid	8270D	283880
580-79910-1 MS	SITK-18-AS-1	SPLP West	Solid	8270D	283880

Lab Chronicle

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-AS-1

Lab Sample ID: 580-79910-1
Date Collected: 08/23/18 13:30
Matrix: Solid
Date Received: 08/25/18 09:20

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	283529	09/08/18 11:30	W1T	TAL SEA
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	284209	09/18/18 15:39	RSB	TAL SEA
SPLP West	Leach	1312			283435	09/06/18 19:38	R1K	TAL SEA
SPLP West	Prep	3510C			283880	09/13/18 10:05	KS	TAL SEA
SPLP West	Analysis	8270D		1	283944	09/13/18 21:40	W1T	TAL SEA

Client Sample ID: SITK-18-TB-AS-1

Lab Sample ID: 580-79910-2
Date Collected: 08/23/18 08:00
Matrix: Solid
Date Received: 08/25/18 09:20

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Prep	5035			283200	09/04/18 18:30	ASJ	TAL SEA
Total/NA	Analysis	8260C		1	283203	09/05/18 04:23	W1T	TAL SEA

Client Sample ID: SITK-18-AS-2

Lab Sample ID: 580-79910-3
Date Collected: 08/23/18 13:45
Matrix: Solid
Date Received: 08/25/18 09:20

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	283529	09/08/18 11:56	W1T	TAL SEA
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	284209	09/18/18 16:04	RSB	TAL SEA
SPLP West	Leach	1312			283435	09/06/18 19:38	R1K	TAL SEA
SPLP West	Prep	3510C			283880	09/13/18 10:05	KS	TAL SEA
SPLP West	Analysis	8270D		1	283944	09/13/18 22:31	W1T	TAL SEA

Client Sample ID: SITK-18-AS-3

Lab Sample ID: 580-79910-4
Date Collected: 08/23/18 14:00
Matrix: Solid
Date Received: 08/25/18 09:20

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	283529	09/08/18 12:22	W1T	TAL SEA
SPLP	Leach	1312			283436	09/06/18 19:56	R1K	TAL SEA
SPLP	Analysis	8260C		100	284209	09/18/18 16:28	RSB	TAL SEA
SPLP West	Leach	1312			283435	09/06/18 19:38	R1K	TAL SEA
SPLP West	Prep	3510C			283880	09/13/18 10:05	KS	TAL SEA
SPLP West	Analysis	8270D		1	283944	09/13/18 22:57	W1T	TAL SEA

Lab Chronicle

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Client Sample ID: SITK-18-PW-1
Date Collected: 08/23/18 14:50
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-5
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	8260C		1	283011	09/01/18 09:28	CJ	TAL SEA
Total/NA	Prep	3520C			282779	08/29/18 12:59	JSM	TAL SEA
Total/NA	Analysis	8270D		1	283155	09/04/18 20:24	T1W	TAL SEA

Client Sample ID: SITK-18-PW-4
Date Collected: 08/23/18 15:15
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-6
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	8260C		1	283011	09/01/18 09:54	CJ	TAL SEA
Total/NA	Prep	3520C			282779	08/29/18 12:59	JSM	TAL SEA
Total/NA	Analysis	8270D		1	283155	09/04/18 20:48	T1W	TAL SEA

Client Sample ID: SITK-18-PW-2
Date Collected: 08/23/18 16:00
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-7
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	8260C		1	283011	09/01/18 10:19	CJ	TAL SEA
Total/NA	Prep	3520C			282779	08/29/18 12:59	JSM	TAL SEA
Total/NA	Analysis	8270D		1	283155	09/04/18 21:13	T1W	TAL SEA

Client Sample ID: SITK-18-PW-3
Date Collected: 08/23/18 16:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-8
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	8260C		1	283011	09/01/18 10:45	CJ	TAL SEA
Total/NA	Prep	3520C			282779	08/29/18 12:59	JSM	TAL SEA
Total/NA	Analysis	8270D		1	283155	09/04/18 21:38	T1W	TAL SEA

Client Sample ID: SITK-18-TB-PW-1
Date Collected: 08/23/18 08:30
Date Received: 08/25/18 09:20

Lab Sample ID: 580-79910-9
Matrix: Water

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Prepared or Analyzed	Analyst	Lab
Total/NA	Analysis	8260C		1	283011	09/01/18 05:07	CJ	TAL SEA

Laboratory References:

TAL SEA = TestAmerica Seattle, 5755 8th Street East, Tacoma, WA 98424, TEL (253)922-2310

Accreditation/Certification Summary

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Laboratory: TestAmerica Seattle

All accreditations/certifications held by this laboratory are listed. Not all accreditations/certifications are applicable to this report.

Authority	Program	EPA Region	Identification Number	Expiration Date
Alaska (UST)	State Program	10	17-024	01-19-19
ANAB	DoD ELAP		L2236	01-19-19
ANAB	ISO/IEC 17025		L2236	01-19-19
California	State Program	9	2901	11-05-18
Montana (UST)	State Program	8	N/A	04-30-20
Nevada	State Program	9	WA000502019-1	07-31-19
Oregon	NELAP	10	WA100007	11-05-18
US Fish & Wildlife	Federal		LE058448-0	07-31-19
USDA	Federal		P330-14-00126	02-10-20
Washington	State Program	10	C553	02-17-19

Sample Summary

Client: Ahtna Engineering Services LLC
Project/Site: Indian River Asphalt Plant-Sitka

TestAmerica Job ID: 580-79910-1

Lab Sample ID	Client Sample ID	Matrix	Collected	Received
580-79910-1	SITK-18-AS-1	Solid	08/23/18 13:30	08/25/18 09:20
580-79910-2	SITK-18-TB-AS-1	Solid	08/23/18 08:00	08/25/18 09:20
580-79910-3	SITK-18-AS-2	Solid	08/23/18 13:45	08/25/18 09:20
580-79910-4	SITK-18-AS-3	Solid	08/23/18 14:00	08/25/18 09:20
580-79910-5	SITK-18-PW-1	Water	08/23/18 14:50	08/25/18 09:20
580-79910-6	SITK-18-PW-4	Water	08/23/18 15:15	08/25/18 09:20
580-79910-7	SITK-18-PW-2	Water	08/23/18 16:00	08/25/18 09:20
580-79910-8	SITK-18-PW-3	Water	08/23/18 16:30	08/25/18 09:20
580-79910-9	SITK-18-TB-PW-1	Water	08/23/18 08:30	08/25/18 09:20

TestAmerica Anchorage
2000 W. International Airport Road
Suite A10
Anchorage, AK 99502
Phone: 907.563.9200 Fax: 907.563.9210

Chain of Custody Record

248885

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Laboratories, Inc.
TAL-8210 (0713)

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

Client Contact	Project Manager: Nino Muniz	Site Contact:	Date:	COC No:
Company Name: AHTNA ENGINEERING SERVICES	Tel/Fax: 907-433-0731	Lab Contact:	Carrier:	1 of 1 COCs
Address: 110 W 38th Ave, Anchorage, AK	Analysis Turnaround Time			Sampler: A. FITZGERALD
City/State/Zip: Anchorage, AK 99503	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
Phone: 907-646-2969	TAT if different from Below			Walk-in Client:
Fax:	☐ 2 weeks			Lab Sampling:
Project Name: Indian River Asphalt Plant	☐ 1 week			Job / SDG No.:
Site: Sitka	☐ 2 days			Loc: 580 79910
P O #	☐ 1 day			

Therm. ID: #2 Cor: 2.9 ° Unc: 2.8 °
Cooler Dsc: Lrg Blue
Packing: Bubble
Cust. Seal: Yes X No
Wet/Packs/Dry Ice/None
IR S: 1.5/1.5
FedEx:
UPS:
Lab Cour: G.S.
Other:

Therm. ID: #2 Cor: 2.9 ° Unc: 2.8 °
Cooler Dsc: Lrg Blue
Packing: Bubble
Cust. Seal: Yes X No
Wet/Packs/Dry Ice/None
IR S: 3.0/3.0
FedEx:
UPS:
Lab Cour: G.S.
Other:

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y/N)	Perform MS / MSD (Y/N)	8260C VOC	8270D SVOC	Sample Specif.
SITK-18-AS-1	8/23/18	1330	G	S	2			X	X	
SITK-18-TB-AS-1		0800		S	1			X		TRIP BLANK
SITK-18-AS-2		1345		S	2			X	X	
SITK-18-AS-3		1400		S	2			X	X	
SITK-18-PW-1		1450		W	5			X	X	
SITK-18-PW-4		1515		W	5			X	X	
SITK-18-PW-2		1600		W	5			X	X	
SITK-18-PW-3		1630		W	5			X	X	
SITK-18-TB-PW-1		0830		W	3			X		TRIP BLANK

580-79910 Chain of Custody

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for_____ Months

Special Instructions/QC Requirements & Comments:
Contact PM for Questions regarding analysis.

Custody Seals Intact: ☐ Yes ☐ No	Custody Seal No.:	Cooler Temp. (°C): Obs'd:	Corr'd:	Therm ID No.:

	Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
1	ALEXA FITZGERALD.	AES	8/24/18 1000		TA-AK	8/24/18 10:00
2		TA AK	8/24/18 12:30			
3				Received in Laboratory by: B. Hall	SEA TA	8.24.18 0920

2.4.2
8.25.18
9/21/2018

Login Sample Receipt Checklist

Client: Ahtna Engineering Services LLC

Job Number: 580-79910-1

Login Number: 79910
List Number: 1
Creator: Hobbs, Kenneth F

List Source: TestAmerica Seattle

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	N/A	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	False	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

(This Page Intentionally Left Blank)

Appendix D – Data Quality Report and ADEC Laboratory Data Review Checklist

(This Page Intentionally Left Blank)

DATA QUALITY REVIEW

Date: November 27, 2018

Project : Indian River Asphalt Plant
Site: Sitka
Laboratory: TestAmerica Laboratories, Inc.
Work Order: 580-79910-1

Reviewer Name: Jess St. Laurent, Ahtna
Reviewer Title: Project Chemist

INTRODUCTION

Table 1 lists the field sample numbers, corresponding laboratory numbers, and identifies quality control (QC) samples.

TABLE 1: FIELD SAMPLE PLAN OVERVIEW

Field Sample ID	Lab Sample ID	Quality Control
SITK-18-AS-1	580-79910-1	Primary
SITK-18-TB-AS-1	580-79910-2	Trip blank
SITK-18-AS-2	580-79910-3	Duplicate
SITK-18-AS-3	580-79910-4	
SITK-18-PW-1	580-79910-5	Primary
SITK-18-PW-4	580-79910-6	Duplicate
SITK-18-PW-2	580-79910-7	
SITK-18-PW-3	580-79910-8	
SITK-18-TB-PW-1	580-79910-9	Trip blank

DATA QUALIFIER DEFINITIONS

For the purpose of this Data Quality Review (DQR) the following code letters and associated definitions are provided for use by the project chemist to summarize the data quality.

R Reported value is "rejected." Resampling or reanalysis may be necessary to verify the presence or absence of the compound.

J The associated numerical value is an estimated quantity because QC criteria were not met, may be biased high or low.

UJ The reported quantitation limit is estimated because QC criteria were not met and the element or compound was not detected.

Q The result is qualified due to quality control criteria not being met

DATA REVIEW

This DQR includes a review, where appropriate, of the following parameters:

- Data completeness
- Chain of Custody (COC) and Cooler Receipt Forms
- Holding times and preservation
- Analytical reporting limits (limits of quantitation [LOQ] and method detection limits [DL])
- Blank analysis results
- Surrogate recoveries (organics only)
- Field duplicates
- Laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) results

Each analysis that was performed is evaluated in the following subsections of this report, and only the criteria exceedances that impact data qualification or require assessment beyond laboratory documentation are discussed.

Validation was conducted in accordance with the USEPA document "*Test Methods for Evaluating Solid Wastes, SW-846, revision 6*" (July 2014 and updates), USEPA *Contract Laboratory Program National Functional Guidelines for Inorganic* (January, 2017) *and Organic* (January, 2017 *Review*, where and when applicable.

Sample Receipt Conditions

Samples were submitted to TestAmerica Laboratories, Inc. in Anchorage, Alaska, and then transferred to their lab in Tacoma, WA. Four water samples and three soil samples, along with one trip blank for each matrix were submitted in one cooler under an intact custody seal. Data was reported in sample delivery group (SDG) 580-79910-1.

Holding Times and Preservatives

All samples were received within the holding times specified by the analytical method. The four water samples, SITK-18-PW-1, SITK-18-PW-4, SITK-18-PW-2 and SITK-18-PW-3 were incorrectly preserved (acidified)for method SW8270D.However, the laboratory did not post acidify the samples and therefore the data usability is not affected.

PRECISION

Field Duplicates

Two duplicate sets were submitted for analysis one for each matrix type. The asphalt duplicate set is primary sample SITK-18-AS-1 and duplicate sample SITK-18-AS-2. The samples for the water set are primary SITK-18-PW-1 and duplicate SITK-18-PW-4. RPDs were calculated using the following equation for the primary and duplicate field samples when both analytes were detected. Calculated RPDs.

EQUATION 1 – RELATIVE PERCENT DIFFERENCE

$$RPD\ (\%) = \text{Absolute Value of: } \frac{(R_1 - R_2)}{((R_1 + R_2)/2)} \times 100$$

Where R_1 = Sample Concentration

R_2 = Field Duplicate Concentration

There were no analytes that were detected in both samples associated with the duplicate sets, no RPDs needed to be calculated. However, Nitrobenzene was detected in the water primary sample SITK-18-PQ-1 and not the duplicate. The analyte in both the primary and duplicate samples should be qualified Q. These samples are qualified "Q" due to poor field duplicate precision.

Matrix Spike/Duplicates, Laboratory Control Samples/Duplicates and Internal Standards

Laboratory RPD errors are detailed below in Table 1.

TABLE 1: LABORATORY PRECISION ERRORS

Error	Data Impact	Qualified Samples
RPD for the LCS and LCSD in method 8270D is outside control limits for 4-Chloroaniline	The associated samples do not contain any detections.	None
RPD for LCS and LCSD in method 8270D is outside control limits for Phenol, Pyrene, Fluoranthene, Bis(2-chloroethyl)ether, 1,2,4-Trichlorobenzene, Isophorone, Acenaphthene, Benzo[g,h,i]perylene, Bis(2-chloroethoxy)methane, 2-Chlorophenol, and Dibenz(a,h)anthracene	The associated samples do not contain any detections.	None

ACCURACY

Matrix Spike/Duplicates, Laboratory Control Samples/Duplicates and Internal Standards

Laboratory errors where %R was above or below the laboratories control samples are noted in Table 2 below.

TABLE 2: LABORATORY ACCURACY ERRORS

Error	Data Impact	Qualified Samples
1,3-Dichloropropane was recovered below control limits	The associated samples do not contain any detections.	None
Benzyl alcohol, 4-Chloroaniline, Hexachlorocyclopentadiene, and Bis(2-ethylhexyl) were recovered outside control limits	The associated samples do not contain any detections.	None
Benzyl alcohol, 4-Chloroaniline, Hexachlorocyclopentadiene, and Bis(2-ethylhexyl) phthalate were recovered outside control limits for the LCS or LCSD	The associated samples do not contain any detections.	None
1,3-Dichlorobenzene, Bis(2-chloroethyl)ether, 2,4-Dinitrophenol, and Butyl benzyl phthalate recovered outside control limits for the LCS or LCSD	The associated samples do not contain any detections.	None

Surrogate Recovery

All surrogate recoveries were within laboratory control limits except for one listed in Table 3 below.

TABLE 3: SURROGATE RECOVERY ERRORS

Error	Data Impact	Qualified Samples
Surrogate 2-Fluorobiphenyl in method 8270D did not meet control criteria.	There are additional passing quality control criteria for samples SITK-18-AS-2 and SITK-18-AS-3	None

REPRESENTATIVENESS

All samples were collected in accordance with the work plan. Samples collected are considered representative of conditions and meet data quality objectives discussed in the work plan.

COMPARABILITY

One laboratory was used and one SDG was received for this project. The results, methods, procedures, quantitation units, and format of the work order are comparable in quality and data validity to all applicable regulations.

COMPLETENESS

All data necessary to complete a level II data validation on this SDG was provided. No data were rejected, and therefore 100% of the results are usable. This exceeds the EPA 85% minimum project completeness goal.

SENSITIVITY

All results were evaluated to the LOD. No qualifications were made based on LODs.

Trip Blanks

Two trip blanks were submitted with the samples. No results were detected above the LOD, the exceptions are in Table 4 below.

TABLE 4: TRIP BLANK ERRORS

Error	Data Impact	Qualified Samples
Bromobenzene was detected is TB SITK-18-TB-AS-1.	The analyte is not detected in any of the samples.	None

Method Blanks

Target analytes were not detected in the method blanks.

OVERALL ASSESSMENT

Based on the data review completed, no data were rejected. Data qualifiers were assigned due to poor field duplicate precision. All analytical data is considered usable for the purpose of evaluating the presence or absence and magnitude of the suspected site contaminants.

(This Page Intentionally Left Blank)

Laboratory Data Review Checklist

Completed by: Jess St.Laurent

Title: Chemist Date: 11/27/18

CS Report Name: Indian River Asphalt Plant-Sitka Report Date: 9/21/18

Consultant Firm: Ahtna Engineering Services

Laboratory Name: TestAmerica Laboratories, Inc. Laboratory Report Number: 580-79910-1

ADEC File Number: 1525.38.055 ADEC RecKey Number:

1. Laboratory
   a. Did an ADEC CS approved laboratory receive and perform all of the submitted sample analyses?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

   b. If the samples were transferred to another "network" laboratory or sub-contracted to an alternate laboratory, was the laboratory performing the analyses ADEC CS approved?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

      Samples were transferred from TestAmerica in Anchorage AK to TestAmerica in Tacoma WA.

2. Chain of Custody (COC)
   a. COC information completed, signed, and dated (including released/received by)?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

   b. Correct analyses requested?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

3. Laboratory Sample Receipt Documentation
   a. Sample/cooler temperature documented and within range at receipt (4° ± 2° C)?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

   b. Sample preservation acceptable – acidified waters, Methanol preserved VOC soil (GRO, BTEX, Volatile Chlorinated Solvents, etc.)?

      The lab states that improper preservation was used for method 8270D samples SITK-18-PW-1, SITK-18-PW-4, SITK-18-PW-2 and SITK-18-PW-3.

☐Yes ☒ No ☐NA (Please explain.) Comments:

c. Sample condition documented – broken, leaking (Methanol), zero headspace (VOC vials)?
☒Yes ☐ No ☐NA (Please explain.) Comments:

The wrong preservative was used for method 8270D in the following samples SITK-18-PW-1, SITK-18-PW-4, SITK-18-PW-2 and SITK-18-PW-3.

d. If there were any discrepancies, were they documented? For example, incorrect sample containers/preservation, sample temperature outside of acceptable range, insufficient or missing samples, etc.?
☒Yes ☐ No ☐NA (Please explain.) Comments:

e. Data quality or usability affected? (Please explain.)
Comments:

Data quality and usability is not affected by the incorrect preservative.

4. Case Narrative
a. Present and understandable?
☒Yes ☐ No ☐NA (Please explain.) Comments:

b. Discrepancies, errors or QC failures identified by the lab?
☐Yes ☐ No ☒NA (Please explain.) Comments:

Discrepancies are documented.

c. Were all corrective actions documented?
☐Yes ☐ No ☒NA (Please explain.) Comments:

d. What is the effect on data quality/usability according to the case narrative?
Comments:

Usability is not affected.

5. Samples Results
a. Correct analyses performed/reported as requested on COC?
☒Yes ☐ No ☐NA (Please explain.) Comments:

b. All applicable holding times met?

☒Yes ☐ No ☐NA (Please explain.) Comments:

c. All soils reported on a dry weight basis?
☒Yes ☐ No ☐NA (Please explain.) Comments:

Yes dry weights were used for the soil samples.

d. Are the reported PQLs less than the Cleanup Level or the minimum required detection level for the project?
☒Yes ☐ No ☐NA (Please explain.) Comments:

e. Data quality or usability affected?
Comments:

Data quality and usability is not affected with respect to the reported sample results.

6. QC Samples
   a. Method Blank
      i. One method blank reported per matrix, analysis and 20 samples?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

      ii. All method blank results less than PQL?
      ☒Yes ☐ No ☐NA (Please explain.) Comments:

      iii. If above PQL, what samples are affected?
      Comments:

      iv. Do the affected sample(s) have data flags and if so, are the data flags clearly defined?
      ☐Yes ☐ No ☒NA (Please explain.) Comments:

There were no affected samples

      v. Data quality or usability affected? (Please explain.)
      Comments:

Data quality and usability was not affected with respect to the reported method blank results.

   b. Laboratory Control Sample/Duplicate (LCS/LCSD)

      i. Organics – One LCS/LCSD reported per matrix, analysis and 20 samples? (LCS/LCSD required per AK methods, LCS required per SW846)

☒Yes ☐ No ☐NA (Please explain.) Comments:

ii. Metals/Inorganics – one LCS and one sample duplicate reported per matrix, analysis and 20 samples?
☐Yes ☐ No ☒NA (Please explain.) Comments:

iii. Accuracy – All percent recoveries (%R) reported and within method or laboratory limits? And project specified DQOs, if applicable. (AK Petroleum methods: AK101 60%-120%, AK102 75%-125%, AK103 60%-120%; all other analyses see the laboratory QC pages)
☐Yes ☒ No ☐NA (Please explain.) Comments:

Several analytes were recovered outside control limits for LCS and LCSD samples.

iv. Precision – All relative percent differences (RPD) reported and less than method or laboratory limits? And project specified DQOs, if applicable. RPD reported from LCS/LCSD, MS/MSD, and or sample/sample duplicate. (AK Petroleum methods 20%; all other analyses see the laboratory QC pages)
☒Yes ☐ No ☐NA (Please explain.) Comments:

The RPDs associated with method 8270D had several analytes not meet control criteria.

v. If %R or RPD is outside of acceptable limits, what samples are affected?
Comments:

No samples were affected because the associated samples do not contain any detections.

vi. Do the affected sample(s) have data flags? If so, are the data flags clearly defined?
☐Yes ☐ No ☒NA (Please explain.) Comments:

vii. Data quality or usability affected? (Use comment box to explain.)
Comments:

Data quality or usability is not affected with respect to the reported results.

c. Surrogates – Organics Only

i. Are surrogate recoveries reported for organic analyses – field, QC and laboratory samples?
☒Yes ☐ No ☐NA (Please explain.) Comments:

ii. Accuracy – All percent recoveries (%R) reported and within method or laboratory limits? And project specified DQOs, if applicable. (AK Petroleum methods 50-150 %R; all other analyses see the laboratory report pages)

The %R for surrogate 2-Fluorobiphenyl in cilient sample SITK-18-AS-2, SITK-18-AS-3 did not meet control criteria.

☐Yes ☒ No ☐NA (Please explain.) Comments:

iii. Do the sample results with failed surrogate recoveries have data flags? If so, are the data flags clearly defined?
☒Yes ☐ No ☐NA (Please explain.) Comments:

Within method 8270D, 2-Fluorobiphenyl, one of six surrogates in client sample SITK-18-AS-2, SITK-18-AS-3 did not meet control criteria.

iv. Data quality or usability affected? (Use the comment box to explain.)
Comments:

Data quality or usability is not affected with regards to the surrogate results.

d. Trip blank – Volatile analyses only (GRO, BTEX, Volatile Chlorinated Solvents, etc.): Water and Soil

i. One trip blank reported per matrix, analysis and for each cooler containing volatile samples? (If not, enter explanation below.)
☒Yes ☐ No ☐NA (Please explain.) Comments:

Two trip blanks were submitted with samples.

ii. Is the cooler used to transport the trip blank and VOA samples clearly indicated on the COC? (If not, a comment explaining why must be entered below)
☒Yes ☐ No ☐NA (Please explain.) Comments:

iii. All results less than PQL?
☐Yes ☒ No ☐NA (Please explain.) Comments:

Analyte Bromobenzene was detected in trip blank SITK-18-TB-AS-1.

iv. If above PQL, what samples are affected?
Comments:

Bromobenzene was not detected in any of the samples, there for the trip blank contamination has no impact.

v. Data quality or usability affected? (Please explain.)
Comments:

Data quality and usability are not affected by the trip blank contamination.

e. Field Duplicate

i. One field duplicate submitted per matrix, analysis and 10 project samples?
☒Yes ☐ No ☐NA (Please explain.) Comments:

Two duplicate sets were submitted to the lab one for each matrix type. The soil samples are primary SITK-18-AS-1 and duplicate sample SITK-18-AS-2. The water samples are primary SITK-18-PW-1 and duplicate SITK-18-PW-4.

ii. Submitted blind to lab?
☒Yes ☐ No ☐NA (Please explain.) Comments:

iii. Precision – All relative percent differences (RPD) less than specified DQOs? (Recommended: 30% water, 50% soil)

RPD (%) = Absolute value of: $\frac{(R_1-R_2)}{((R_1+R_2)/2)} \times 100$

Where R_1 = Sample Concentration
R_2 = Field Duplicate Concentration

☒Yes ☐ No ☐NA (Please explain.) Comments:

All calculated samples are within the recommended limits.

iv. Data quality or usability affected? (Use the comment box to explain why or why not.)

Comments:

Nitrobenzene was detected in the water primary, sample SITK-18-PW-1. Both primary and duplicate samples were qualifed "Q" due to poor precision. Data quality or usability is not affected but qualified results should be considered estimated.

f. Decontamination or Equipment Blank (If not used explain why).

☐Yes ☐ No ☒NA (Please explain.) Comments:

No equipment blank was submitted. Disposable sampling equipment was used.

i. All results less than PQL?

☒Yes ☐ No ☐NA (Please explain.) Comments:

ii. If above PQL, what samples are affected?

Comments:

iii. Data quality or usability affected? (Please explain.)

Comments:

Usability is not affected.

7. Other Data Flags/Qualifiers (ACOE, AFCEE, Lab Specific, etc.)
   a. Defined and appropriate?
      ☐ Yes ☐ No ☐ NA (Please explain.) Comments:

No additional data qualifiers were used.

(This Page Intentionally Left Blank)

Appendix E – Sampling and Analysis Plan

(This Page Intentionally Left Blank)

Sampling and Analysis Plan

Sitka National Historical Park

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
EDL Number 5AKR2141

Prepared by

Michael Baker International/Ahtna Engineering Services, LLC

8/10/2018

Herminio (Nino) Muniz, PG	**William F. Heubner**
Project Manager	NPS Project Engineer
August 10, 2018	August 10, 2018
Date	Date

By signing above, the signatories verify that they understand and concur with the information, procedures, and recommendations presented herein

Table of Contents

Appendices

List of Figures

List of Tables

List of Abbreviations and Acronyms

AAC	Alaska Administrative Code
ADEC	Alaska Department of Environmental Conservation
Ahtna	Ahtna Engineering Services, LLC
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cfs	cubic feet per second
COC	Chain of Custody
CSM	Conceptual Site Model
DQO	Data Quality Objective
DRO	diesel range organics
EDL	Environmental and Disposal Liabilities
FSP	Field Sampling Plan
GPS	Global Positioning System
IDW	Investigative-derived waste
IDQTF	Intergovernmental Data Quality Task Force
LCS	laboratory control samples
LCSD	laboratory control samples with duplicates
LOQ	limit of quantitation
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
ml	Milliliter
MS/MSD	matrix spike/matrix spike duplicate
NCP	National Oil and Hazardous Substances Pollution Contingency Plan (AKA, National Contingency Plan)
NPS	National Park Service
PA	Preliminary Assessment
PM	Project Manager
ppm	parts per million
PPE	Personal protective equipment
PQL	Practical quantitation limit
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control

RL	reporting limit
RPD	Relative percent difference
RRO	residual range organics
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SI	Site Inspection
SITK	Sitka National Historical Park
SOP	Standard Operating Procedure
SPLP	simulated precipitation leaching procedure
SVOC	semi volatile organic compounds
TAH	total aromatic hydrocarbons
TAqH	total aqueous hydrocarbons
TCLP	Toxicity characteristic leaching procedure
TPH	total recoverable petroleum hydrocarbons
USC	United States Code
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
VOC	volatile organic compounds
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
°C	Degrees Celsius
°F	Degrees Fahrenheit

1 Introduction

This document serves as the Sampling and Analysis Plan (SAP) for the Indian River Asphalt Plant Preliminary Assessment (PA)/Site Inspection (SI) in the Sitka National Historical Park (SITK), Alaska. The work is being conducted under NPS notice-to-proceed number 140P9718Q0026.

The site is a former asphalt hot plant located on the northeast bank of the Indian River, where surplus asphalt and debris from the plant were buried. Significant storm events and river flooding in recent years have given way to erosion in the area, exposing the buried materials. Reports of sheen on the river, likely associated with this site, have prompted investigation of the potential environmental concerns (NPS 2018).

The purpose of this SAP is to define:

- The potential for petroleum impacts from the remaining asphalt in the ground at the Indian River Asphalt Plant;
- Whether compounds from the asphalt are or are not currently impacting the waters of the Indian River;
- The data quality objectives (DQO) that will ensure the amount of data collected is sufficient and that the quality of data meets the project needs; and
- The methods that will be used to collect site and analytical data.

1.1 Regulatory Authority

This SAP was generated in accordance with the United States Environmental Protection Agency's (USEPA) *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA 2006a), *Guidance for Quality Assurance Project Plans* (USEPA 2002a), *EPA Requirements for Quality Assurance Project Plans* (USEPA 2001), and the Intergovernmental Data Quality Task Force's (IDQTF) *Uniform Federal Policy for Quality Assurance Project Plans* (IDQTF 2005). The National Park Service (NPS) is authorized under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. §§ 9601 et seq., to respond as the Lead Agency to a release or threatened release of hazardous substances and/or a release or threatened release of any pollutant or contaminant that may present an imminent and substantial danger to public health or welfare on NPS land. For this project, the NPS will defer to the Alaska Department of Environmental Conservation (ADEC) for primary oversight. The regulations covering this site are under Title 18 of the Alaska Administrative Code Chapter 75 (18 AAC 75).

CERCLA's implementing regulations, codified in the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 CFR Part 300, establishes the framework for responding to such releases and threatened releases. The NCP prescribes two similar processes for responding to releases: removal actions and remedial actions (See NCP Sections 300.400 through 300.440). If environmental samples are to be collected under either process, a SAP is required (See NCP Sections 300.415 and 300.430). The SAP is comprised of two parts: the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP). The FSP describes the number, types, and locations of samples as well as the types of analyses that will

be conducted on the samples. The QAPP describes the project's policy, organization, and functional activities as well as the DQOs, and measures necessary to achieve the goals of the study.

In addition, the NPS has a number of regulations that apply to the release of hazardous substances on NPS land (see NPS 2014) including the NPS Organic Act of 1916 (16 USC §1, et seq. 36 CFR Part 1), which requires that the NPS manage parks in order to conserve the scenery, natural and historic objects, and wildlife and to provide for their enjoyment by such means as will leave them unimpaired for the enjoyment of future generations. Therefore, whether the Site poses risks to the interaction of organisms and the environment is especially relevant to the NPS responsibility to protect park resources.

1.2 Purpose of Field Sampling

The purpose of this sampling event is to assess whether, as the Indian River erodes the former asphalt plant area, the petroleum constituents in the remaining asphalt in the ground is currently impacting the waters of the Indian River, and to evaluate the potential for the asphalt to impact the soil and groundwater in the future.

One sampling event will be conducted in August 2018, during a period of lower flow in the Indian River. The NPS will use data collected during this field investigation to support potential response actions that may be undertaken by the NPS or other parties. Data generated from these sampling events will be used in accordance with the provisions outlined in the DQOs. The following data will be collected during the sampling events:

- Collection of pore water samples to assess volatile organic compounds (VOC), and semi-volatile organic compounds (SVOC) for assessment of stream impacts.
- Collection of asphalt samples from the ground surface. The asphalt samples will be subjected to simulated precipitation leaching procedures (SPLP) and the leachate analyzed for VOC and SVOC petroleum constituents.

The NPS will use the data obtained from these investigations in accordance with the provisions outlined in the DQOs detailed in Section 4. The SAP will support the PA/SI.

1.3 Site Location

The Indian River Asphalt Plant site is located within the boundaries of SITK, in Sitka (Figure 1), Alaska. Detailed location information is listed below:

- Indian River Asphalt Plant, Sitka National Historical Park,
  103 Monastery Street,
  Sitka, Alaska, 99853
- Site Environmental and Disposal Liabilities (EDL) number: 5AKR2141
- The latitude/longitude of the site is 57 degrees, 2 minutes, 50 seconds North, 135 degrees, 18 minutes, 39 seconds East
- Northwest quarter of Section 6, Township 56 South, Range 64 East, Copper River Meridian

2 Site Description, Previous Investigations, and Conceptual Site Model

This section summarizes all the known environmental information and historical activities that have occurred at the Indian River Asphalt Plant site, and presents this information in the form of a graphical Conceptual Site Model (CSM). The development of a clear and thorough CSM is a critical component for ensuring that key site elements are considered before any samples are collected, gaining stakeholder approval, and assisting the Planning Team in developing the DQOs (Section 4), as well as assisting the field team in making decisions in the field. Figure 3 is a graphical CSM that illustrates the information detailed in the following sections.

2.1 Key Site Features

2.1.1 Site Description

The Indian River Asphalt Plant site is located on the northeast bank of the Indian River, that encompasses an area of approximately 3/4 acre within the boundary of SITK (Figure 2). The site is bordered by Park lands, Sawmill Creek Road to the north, Crescent Bay to the south, Arrowhead Trailer Court to the east and tidal flats of the Indian River to the west. There are residential, industrial and undeveloped areas surrounding the Park (Shannon&Wilson, 1995). There is an established trail system that runs through the Park and site as well as a memorial plaque of the 1804 Battle of Sitka, between the Tlingit tribe in Sitka and the Russian explorers, which receives visitors regularly. The SITK NPS Visitor Center offers the public guided walking tours to the memorial from May through September.

The nearest residential structures are the homes in the Arrowhead Trailer Court, and there is a wood fence separating the Park from the Arrowhead Trailer Court homes. Access to the site can be gained via a trail off of Sawmill Creek Road, the footbridge that spans the Indian River, or at the southern end of the fenced-off Arrowhead Trailer Court residential area.

Operational History

Beginning in the 1940s, gravel was extracted from the Indian River by private interests as well as the U.S. Army and Navy. In the late 1950s, the Indian River asphalt plant was established and operated by Morrison-Knudsen Company to build roadways in the city of Sitka. The plant used gravel from the river as aggregate to produce the pavement. An area 60-feet deep was dredged at the mouth of the Indian River, and approximately 1.5 million cubic yards of material was dredged from the sea. After several years of operation, the plant was closed and debris that was left at the site, such as machinery, metal, cable, and old asphalt, was buried at the site and abandoned (Shannon&Wilson, 1995). The site is mostly overgrown with forest since that time, however erosion during storm events has exposed the buried debris associated with the former asphalt plant (NPS, 2018).

Reports of a sheen in the Indian River at the base of this site have been documented since 1990. Results from soil samples taken from the river bank at the site taken in 1993 and again in 1995, indicate diesel range organic (DRO) and residual range organic (RRO) exceedances of ADEC Method Two migration to groundwater soil cleanup levels. The analytical results of samples taken for VOC did not exceed cleanup levels. While sample results did detect the presence of mercury, arsenic, chromium, and lead, they were reportedly not significantly higher than background levels. Remedial alternatives were investigated for

the site. However, excessive cost and disruption to the park and potentially marine environment resulted in a decision to conduct monitoring of the site's water quality rather than initiate a full remedial effort. Results of the water quality monitoring between 2002 and 2005 showed no significant contamination concentrations, and the water quality monitoring was halted after an agreement made between ADEC and NPS (NPS, 2018).

Increased erosion from storm activity in recent years has uncovered more debris from the site, including large patches of old tar and rusted metal debris. Sheen on the river water was also once again noted by the NPS Resource Manager in Sitka, however it is unknown at this time if the sheen is the result of biological matter breakdown or petroleum constituents. In 2017, the NPS Alaska Regional Environmental Management Program Coordinator conducted a site visit, surveyed the area and collected two samples of oozing tar found on the beach. The two tar samples were analyzed by a certified laboratory for SVOC, and the results indicated exceedances of ADEC migration to groundwater cleanup levels for several of the SVOC constituents. The large mass of tar was removed the following month by NPS personnel and transported off site for disposal. Several small pieces of tar remain exposed at the site (NPS 2018).

Waste Characteristics

Shannon & Wilson, the consultant who conducted the Environmental Site Assessment in 1995 on behalf of NPS, concluded that there existed at that time approximately 400 cubic yards of DRO and RRO contaminated soil at this site. Contaminants of concern at the site for purposes of the 2018 PA/SI are VOCs and SVOCs.

Site Geology and Hydrogeology

At the head of Indian River and its tributaries are the steep mountain sideslopes and cirque walls, formed during local alpine glaciation, that shed the source rocks for surficial deposits. The deposits consist of primarily graywacke, schist, and phyllite, which cover the majority of the Park, and include alluvium on Indian River's floodplain, estuary, and stream terrace. Ablation till exists on the lateral moraine, and beach sands and gravel are the primary geology on the uplifted beach and uplifted beach meadow where the project site is located (NPS 2015a).

The Indian River forms an estuary as it enters into the ocean just below the site, both of which are considered primary resources of SITK. North of the Park are the Baranof Island mountains, where the Indian River originates. Form here it flows into Sitka Sound, between Crescent and Jamestown Bays. A large U-shaped post-glacial valley watershed exists here, encompassing approximately 12.3 square miles with an elevation range of sea level to 3,800 feet. The river and upper basin alpine regions drain into the watershed toward the ocean. The valley floor consists of a wide, flat area, covered by muskeg and a forest of primarily Sitka spruce and western hemlock (NPS 2016). The project site is densely populated with alder, Sitka spruce and undergrowth (Shannon&Wilson 1995).

Site Hydrology

Characterized by steep topography, the Indian River watershed consists of well-drained shallow soils, and high drainage density. The watershed has a rapid response to rainstorms, which can cause large daily fluctuations in stream flow. Hydrologic calculations indicate that the height of runoff occurs within six hours of a storm event and nearly all rainfall runs through the watershed within 12 to 24 hours. River flow ranges from approximately 20 cubic feet per second (cfs) to 6400 cfs, and it is considered a 100-year flood zone. River discharge is generally at its peak in September and October, tending to decline

throughout winter and early spring. Snowmelt at high elevations results in moderate flow increases in May and June, while minimum flows are most common in December, March, and July (NPS 2016).

Local drinking water for the City of Sika is supplied by surface water from Blue Lake. The Indian River was once considered a drinking water supply for the City, however it is currently no longer used (City and Borough of Sitka 2015).

Local Climate

According to the U.S. Climate Data, Sitka's average annual high temperature is 49.9 degrees Fahrenheit (ºF), and its average annual low temperature is 40.6 ºF. Average annual precipitation from rainfall in the Sitka region is 86.72 inches, and average annual snowfall is 33 inches.

Sensitive Environments

The project site is located within a national park, therefore, the entire area is considered a sensitive environment. Of particular interest, the area has runs of anadromous fish (salmon) along with resident fish (e.g. dolly varden). It is home to many species of birds including bald eagles and small and large mammals.

2.2 Summary of Previous Investigations

2.2.1 Data Quality/Usability

In May of 1993, Ms. Barbara Reilly of the U.S. Army Corps of Engineers collected one soil sample from the site and submitted it for DRO, GRO, total recoverable petroleum hydrocarbons (TPH) and toxicity characteristic leaching procedure (TCLP) for VOCs and TCLP for SVOCs. Results indicated DRO at a level of 1,820 parts per million (ppm) and TPH at 3,400 ppm, which exceed ADEC cleanup levels. No other results exceeded cleanup levels (Shannon&Wilson 1995).

Shannon and Wilson completed a Level 2 Environmental Site Assessment in April 1995 at the former Indian River Asphalt Plant, "*to identify and characterize, to the extent possible, the source of the oil saturated sediment layer and vegetative mat that have been exposed from an eroding bank along the Indian River, and to outline remedial actions for the site*" (Shannon&Wilson 1995). During this investigation, ten test pits were dug at the site and 33 soil samples were collected for DRO, RRO and VOC analysis. Samples collected from the exposed bank were also analyzed for arsenic, cadmium, chromium, lead and mercury.

DRO was detected in 24 of the 33 soil samples, with concentrations ranging from 12 mg/kg to 6,100 mg/kg, with 6 samples exceeding the Method Two migration to groundwater soil cleanup levels of 230 mg/kg. RRO was detected in 27 of the 33 soil samples, with concentrations ranging from 25 mg/kg to 8,700 mg/kg. None of the samples exceeded cleanup levels for VOC constituents, though one sample had an ethylbenzene concentration of 0.12 mg/kg. Arsenic, chromium and lead were reported in all of the 11 analyzed test pit samples with concentrations of 3.5 mg/kg to 12 mg/kg arsenic, 15 mg/kg to 39 mg/kg chromium and 6.3 mg/kg to 3,500 mg/kg lead (the elevated lead reading of 3,500 mg/kg appeared to be limited in quantity and only in one sample in one test pit.). There was one detection of mercury at 0.12 mg/kg. For the two samples collected from the bank, arsenic was detected at 6.3 mg/kg and 26 mg/kg; chromium at 33 mg/kg and 39 mg/kg and lead at 5.6 mg/kg and 10 mg/kg. (NPS 2018). The metals results were found to be consistent with background levels.

The level of exceedances around the site resulted in the opinion that a remedial effort for the removal of contaminated DRO at the site would consist of excavating and disposing of approximately 400 cubic yards of soil and debris. Due to the high cost and potential damage to this area of the park, it was decided that it would be in the best interest of the SITK and State to conduct monitoring of the site rather than a removal effort. Water quality monitoring efforts conducted at the site in the early 2000s detected no significant contamination, and in 2006, the NPS and ADEC agreed to stop monitoring at the site.

Much of the site, approximately 50 feet of beach area, has eroded into the waters of the Indian River and estuary since 1995 and buried debris at the site has become exposed (NPS 2018). In August 2017, the NPS Alaska Region's Environmental Management Program Coordinator conducted a site visit and collected two samples of exposed asphalt tar which was oozing from a large mass on the beach. The samples were sent to a certified laboratory for SVOC analysis, the results of which indicated exceedances of ADEC Method Two migration to groundwater for several of the SVOC constituents. The large mass was removed by NPS personnel and disposed of offsite in September in 2017. Small pieces of asphalt remain at the site, and are exposed periodically at times of high tide and high river events.

2.2.2 Preliminary Identification of Data Gaps

The primary data gaps are as follows:

- The current state of contamination
- Potential impacts to ground and surface water

Soil information from previous assessments, combined with data collected during this investigation, will be used to close these data gaps.

2.2.3 Contaminants of Potential Concern

The site was a former asphalt plant where unused asphalt and other debris associated with asphalt manufacturing were buried after the plant's removal. The contaminants of potential concern are petroleum hydrocarbons associated with the tar used in asphalt including VOC, SVOC, GRO, DRO, and RRO.

2.2.4 Media of Potential Concern

The media of potential concern at this site are soil, groundwater and surface water.

2.3 Current and Future Property Use Scenarios

The site is currently used by visitors of SITK for recreation purposes, by walking the trails within the park that run through the site. Potential human receptors are visitors to the Park, the NPS personnel who work there and maintain the area, and residents that live in the neighboring Arrowhead Trailer Court.

Potential ecological receptors include birds nesting in the area, fish living in or traveling through potentially impacted waters of the Indian River and estuary, mammals crossing the site or consuming plants and animals living within the impacted area that may be exposed to contaminants.

The primary potential human exposure routes include direct contact by touching the exposed tar or by drinking or swimming in the river water. The ingestion of groundwater pathway by humans is considered complete but at this time insignificant as no drinking water well exists in this immediate area (nor will be

installed in the foreseeable future). The City of Sitka water supply source is surface water from Blue Lake.

For wildlife the primary exposure route would also be direct contact of animals burrowing or digging into the impacted soils, or walking across tar on the beach area. Exposure through ingestion could also occur through consumption of plants and animals living with the impacted zone, such as wild plants, fish, birds or mammals that are gathered/hunted for subsistence.

2.4 Graphical Conceptual Site Model

Figure 3 presents the CSM for Indian River Asphalt Plant site. Figure 3 provides a model for the site, showing representations of the potentially impacted soil layers, the potential transport of contaminants from the site through precipitation/leaching to the groundwater and into the Indian River, and the potential transport of contaminants to birds, small mammals, and plants.

2.4.1 Key CSM Assumptions

The following assumptions were made in preparation of the graphical CSM:

- The site is approximately 8 feet above mean sea level, sloping gradually down to the river
- Plant roots extend into the impacted zone
- Invertebrates and insects preyed upon by small mammals are living in the impacted soils
- Unconfined groundwater is present beneath the site at just above the level of the river
- Groundwater flow is to the south, toward the river

(This Page Intentionally Left Blank)

3 DQO Planning Team and Stakeholders

3.1 DQO Planning Team

TABLE 3-1: DQO PLANNING TEAM

Team Members	Responsible Entity	Contact Information	Roles and Responsibilities in DQO process
Bill Heubner	NPS Project Manager (PM)	907-644-3384	The NPS PM ensuring the needs and goals of the NPS are met.
Nino Muniz, PG	Ahtna Project Manager	907-433-0731	The Ahtna PM will provide the analytical and hydrogeological information or the NPS PM to allow him to achieve the NPS needs and goals.
Emily Freitas	Ahtna Chemist	907-433-0725	The Ahtna Chemist will help establish laboratory and field quality control criteria and project action limits. The chemist will communicate any data validation issues and data review corrective actions with the Ahtna PM and the laboratory PM.

3.2 Decision Maker

The decision maker has the ultimate authority based on the recommendations of the DQO Planning Team. The decision maker for this project is:

- Bill Heubner – NPS Project Manager

3.3 Stakeholders

Stakeholders are parties who may be affected by the results of the investigation and/or persons who may later use the data resulting from the DQO process. The stakeholders for this site include:

- The NPS

(This Page Intentionally Left Blank)

4 Data Quality Objectives

4.1 State the Problem

This section defines the problem requiring investigation.

- The former Indian River Asphalt Plant was removed in the late 1950s/early 1960s, and unused asphalt and other metal debris were buried at the site. Analytical results from previous investigations in the 1990s and 2000s indicate the presence of contaminants associated with the buried debris, which have the potential to leach contamination into the site soil, groundwater and bordering Indian River. This PA/SI and subsequent collection of asphalt and pore water samples will provide results showing the level of petroleum impacts (if any) from the remaining asphalt in the ground at the Indian River Asphalt Plant, whether or not it is currently impacting the waters of the Indian River, and whether or not it may impact the soil or groundwater in the future.
    - The primary concern for the site is direct contact of contaminants with SITK visitors, NPS personnel and wildlife.

4.2 Identify the Goal of the Investigation

The goal of this PA/SI is to determine the current status of the contamination at the project site, and whether or not there is a risk for future contamination. With this information, a SI report will be written and recommendations for remedial action, continued monitoring or no action, can be made.

4.2.1 Principal Investigation Question

The principal investigation question is the following:

Is the buried debris remaining at the Indian River Asphalt plant leaching and causing current contamination to the area's soils or groundwaters, or is there potential for it to do so in the future? If so, is the contamination currently posing an unacceptable risk to human health and ecological receptors, or could it in the future? The primary receptors are as follows:

- SITK visitors/recreational users
- NPS personnel accessing the site
- Nearby residents
- Vegetation
- Invertebrates in the soil
- Birds and small mammals ingesting invertebrates or plants
- Fish that may encounter contaminated surface water
- Large mammals ingesting fish, birds, other mammals or plants

4.3 Identify Information Inputs

4.3.1 Previous Data Usability

In May of 1993, DRO at a level of 1,820 ppm and TPH at 3,400 ppm, which exceed ADEC cleanup levels (Shannon&Wilson 1995).

In a Level 2 Environmental Site Assessment conducted in 1995, ten test pits were dug at the site and 33 soil samples were collected for DRO, RRO and VOC analysis. Samples collected from the exposed bank were also analyzed for arsenic, cadmium, chromium, lead and mercury. DRO was detected in 24 of the 33 soil samples, with concentrations ranging from 12 mg/kg to 6,100 mg/kg, with 6 samples exceeding the current Method Two migration to groundwater soil cleanup levels of 230 mg/kg. RRO was detected in 27 of the 33 soil samples, with concentrations ranging from 25 mg/kg to 8,700 mg/kg. None of the samples exceeded cleanup levels for VOC constituents, though one sample had an ethylbenzene concentration of 0.12 mg/kg. Arsenic, chromium and lead were reported in all of the 11 analyzed test pit samples with concentrations of 3.5 mg/kg to 12 mg/kg arsenic, 15 mg/kg to 39 mg/kg chromium and 6.3 mg/kg to 3,500 mg/kg lead (the elevated lead reading of 3,500 appeared to be limited in quantity and only in one sample in one test pit.). There was one detection of mercury at 0.12 mg/kg. For the two samples collected from the bank, arsenic was detected at 6.3 mg/kg and 26 mg/kg; chromium at 33 mg/kg and 39 mg/kg and lead at 5.6 mg/kg and 10 mg/kg. (NPS 2018). The metals results were determined to be consistent with background levels.

In 2017, two samples of asphalt tar were collected and analyzed for SVOC. Several constituents exceed current cleanup levels. SVOC Compounds exceeding current ADEC soil cleanup levels are summarized in Table 4-1:

TABLE 4-1: ASPHALT SVOC EXCEEDENCES

Analyte	Maximum Detected Conc. in mg/kg	ADEC Cleanup Level in mg/kg[1]
Benzo[a]anthracene	1.3	0.28
2,4-Dinitrotoluene	7.7	0.024
Dibenzofuran	5.7	0.97
1-Methylnaphthalene	37	0.41
2-Methylnaphthalene	48	1.3
Naphthalene	21	0.038
Phenanthrene	47	39

Notes:

[1] 18 AAC 75, Table B1, migration to groundwater criteria (November 2017)

ADEC Alaska Department of Environmental Conservation

mg/kg milligrams per kilogram

4.3.2 Data to be Collected in the Current Investigation

The new data required to answer the principal investigation questions are as follows:

- Two samples of asphalt will be collected, extracted using SPLP, and analyzed for the following:
  - VOC
  - SVOC
- Three pore water sample will be collected and analyzed for the following:
  - VOC (total aromatic hydrocarbons [TAH] will be calculated)
  - SVOC (total aqueous hydrocarbons [TAqH] will be calculated)
- Survey to obtain global positioning system (GPS) coordinates of sample locations

Table 4-2 shows the specific requirements for the sampling event to be conducted August 2018 and how each type of data will be used. The FSP is detailed in Section 5.

Table 4-2: Soil Project Sampling Requirements

Sample Type	Sample Use
Asphalt	Assessment of potential to leach to soil and/or groundwater
Pore Water	Assessment of contamination concentrations

- One Ahtna scientist will mobilize to the site in late August 2018 to collect data.
- Collect two asphalt samples from areas where it is exposed on the ground surface within the site boundary and submit them for laboratory for extraction by SPLP and analyses of VOC and SVOC to evaluate the potential for the asphalt to impact soil and groundwater.
- Collect three pore water samples adjacent to the Indian River (upstream, next to and downstream) and submit them for laboratory analyses of VOC and SVOC. Use the data to calculate TAH and TAqH to assess surface water impacts.

4.4 Define the Boundaries of the Investigation

4.4.1 Spatial Boundaries

The boundary of the investigation will consist of samples taken from exposed asphalt tar on the beach and pore water samples taken along the edge of the Indian River, from locations above the site, at the site and below the site.

4.4.2 Temporal Boundaries

The SI and sample collection will take place in August 2018. One sampling event will take place.

4.4.3 Sampling Units

The sample units for this project are defined as:

- Two asphalt samples
- Three pore water samples collected with a pore water sampler and peristaltic pump.

4.4.4 Decision Units

There is only one decision uniot for this site. The decision unit for this site is defined as the former Indian River Asphalt Plant site.

4.5 Develop the Analytic Approach

4.5.1 Decision or Estimation Parameters

The population of this project is all of the samples collected at the site. ADEC Method Two Cleanup levels and surface water cleanup levels will be used to evaluate the analytical results of the samples collected.

4.5.2 Action Levels

The ADEC Method Two Cleanup levels and surface water cleanup levels will define the action level for this site.

4.6 Performance or Acceptance Criteria

4.6.1 Quality Assurance/Quality Control

Quality control (QC) samples are collected in the field to assess the level and quality of sampling and laboratory analysis. Quality assurance (QA) samples are collected in the field to assess the consistency of sampling and laboratory analysis.

A summary of QC/QA samples is depicted in Table 4-3.

Table 4-3: Field QC/QA Samples

QC Samples	Media	Analysis Method	Frequency
Field Duplicate	Aqueous	Same as primary samples	1 per 10 samples (10%) for matrix and each analytical method
Trip Blank	Aqueous	VOC	1 per sample cooler
Temperature Blank	500-mL vials of tap water	--	1 per sample cooler

Key:
% percent
mL milliliter

Field Quality Assurance/Quality Control

Duplicate QC samples will be collected at a rate of 10% of project samples. Duplicate samples will be collected from the location of the suspected highest contamination, anticipated to be the location with the highest field screening result or the most likely soil type and depth for contamination in the event of non-detect field screening results and will be collected exactly the same as primary samples.

No equipment blanks will be necessary as all samples will be collected using disposable sampling equipment. Standard Operating Procedure (SOP) 10 in Appendix A details the collection of field QC samples.

The collection, labeling, and handling procedures will be consistent with those used for the primary samples and QC duplicate samples. The QC/QA samples will be named in the same manner as the primary and duplicate samples, with the next sequential number.

Pickup of QA sampling supplies and delivery of the QA samples will be coordinated by Ahtna.

A summary of QC/QA samples is depicted in Table 4-2.

Samples will be labelled, packaged, and shipped in accordance with the SOPs in Appendix A. Chain of Custody (COC) procedures will be followed as outlined in the SOP included in Appendix A. Note that for this project, one COC will be required per cooler. An example COC form is provided along with other field forms in Appendix D.

Samples will be submitted on a turn-around-time of 21 calendar days to the ADEC-approved and ELAP-certified laboratory, anticipated to be a TestAmerica laboratory. A sample receipt form will be prepared by the laboratory and emailed to the NPS within 24 hours of receipt.

Field duplicates will be collected and labeled following the same sample naming technique described in section 5.2.1.

This section describes the QA/QC procedures that analytical laboratory personnel will follow. Reference the laboratory's QA Plan and the SOPs that the laboratory will follow when preparing and analyzing the samples.

Decontamination Procedures

All non-disposable sampling equipment will be washed in anionic solution, such as Alconox®, and then rinsed with deionized water over a bucket. All other sampling equipment and personal protective equipment (PPE) will be disposable and will not require decontamination. Decontamination will occur between sampling sites and at the end of each field sampling day. See section 5.4 below for additional information.

Instrument/Equipment Testing, Inspection, and Maintenance

Maintenance schedules for each piece of equipment used in the field will comply with the Instrument/Equipment Maintenance SOP (Appendix A). The field manager or Ahtna field scientist will be responsible for ensuring that spare parts for field equipment are available at the project site to minimize equipment downtime, as well as maintenance schedules for each piece of equipment used.

Instrument/Equipment Calibration and Frequency

Instrument calibration will be not be necessary for this sampling event.

Inspection/Acceptance of Supplies and Consumables

Prior to commencing fieldwork, field team members will refer to the work plan and verify that all necessary equipment and materials have been obtained for the project.

Special Training and Certification

No special training or certification will be necessary to complete the project objectives. All field sampling personnel are ADEC "qualified environmental professional" as defined in 18 AAC 75.333.

Laboratory Quality Assurance/Quality Control

TestAmerica is an ADEC-approved and Environmental Laboratory Accreditation Program (ELAP) certified laboratory that is anticipated to perform all analytical analyses. ADEC and ELAP laboratory certifications can be obtained at any time by contacting the lab and are available in Appendix B.

Laboratory Quality Control Samples

SOP created by the laboratory will be used for all analyses required for this project. The SOPs are maintained by the laboratory and will be included as part of the final document. They detail the measurement performance criteria, detection and quantitation limits, calibration procedures and requirements, maintenance, testing, inspections, and corrective actions. The SOPs also detail the internal laboratory QC criteria for each analysis.

Laboratory QC checks shall include at a minimum, method blanks, laboratory control samples (LCS) and laboratory control samples with duplicates (LCSD). Method blanks, LCS, and LCSDs will be included at a rate of at least one per batch or one for every 20 samples.

The laboratory will report the results for all QC samples along with the quantitation limit, the percent recovery, and the relative percent difference (RPD) for duplicates. These QC data will be reviewed by the laboratory and the project chemist to ensure that all results are within the lab-specified acceptable QC

ranges. All blanks should have results less than the quantitation limit, which shall be less than the project screening limits depicted in Table 4-2.

The laboratory SOPs and tables depicting the QC criteria and measures for the methods requested are included in Appendix B.

The contact information for TestAmerica is as follows:

TestAmerica – Anchorage (for sample kit pickup, sample drop-off, and transport of samples to the Seattle location for analysis)

2000 West International Airport Road, Suite A10
Anchorage, Alaska 99502
(907) 563-9200

TestAmerica – Seattle (for sample analysis)

5755 8th Street East
Tacoma, WA 98424

Data Quality Indicators Table

Table 4-4 summarizes the goals of each of the data assessment measurements.

Table 4-4: Data Assessment Goals

Quality Indicator	Goal
Precision	(RPD ≤ method-specific limits for laboratory QC samples, RPD ≤ 50 percent (%) for field duplicates in soil.
Accuracy	All surrogate and lab QC sample percent recoveries are within laboratory limits.
Representativeness	All samples are collected from locations of target field screening readings and in accordance with the approved work plan.
Comparability	All samples analyzed by the same method and SOPs in the laboratory and reported with consistent units. Screening data correlates to analytical data. All samples will be collected and handled in the same way.
Completeness	At least 95% of all samples collected are analyzed, reported, and results are usable. All holding times are met. Cooler temperatures are within 2-6 degrees Celsius (°C).
Sensitivity	All quantitation limits are less than the project target levels. All field and laboratory blanks have results that are less than the quantitation limits and target levels.

4.6.2 Decision Error Limits and Uncertainty Evaluation

Laboratory reporting limits and recovery percentages are presented in Appendix B. All results will be evaluated to the reporting limit (RL) unless the RL is above the project screening levels. In these instances, results will be evaluated to the method detection limit or limit of quantitation (LOQ). The level of uncertainty varies between these laboratory limits but is still 99% confidence and suitable for the purpose of this project. Limits are established prior to the laboratory receipt of the samples but may be adjusted based on possible dilutions performed.

4.6.3 Data Validation and Usability

Data Verification

Initially upon completion of field activities, receipt of all documentation will be completed. All field documents, along with laboratory deliverables, will be collected and evaluated to ensure completeness, accuracy, and accordance with this SAP, the SOPs, and NPS guidelines.

Data Validation

Two types of data will be validated for quality. Screening data will be assessed for quality based on calibration records and comparability to analytical data. Analytical data will be assessed for quality using this SAP, laboratory SOPs, and the USEPA National Functional Guidelines.

Following verification of data deliverables, Ahtna will perform an assessment of data quality on all laboratory data.

For field duplicate samples submitted to the laboratory, RPDs will be calculated and assessed against the project precision data quality indicator of less than 50% for soil samples. As necessary, data qualifiers will be assigned. The data qualifiers used are shown in Table 4-5 below:

TABLE 4-5: DATA QUALIFIERS

Qualifier	Definition
J	Analyte result is considered an estimated value because the level is below the laboratory LOQ but above the detection limit (formerly method detection limit).
B	Analyte result is considered a high estimated value due to contamination present in the method or trip blank.
QH, QL, QN	Analyte result is considered an estimated value biased (high, low, uncertain) due to a quality control failure.
R	Analyte result is rejected – result is not usable. Note that "R" replaces the chemical result (no result shall be reported with an "R" flag.)

The data quality indicator criteria will be measured as precision, accuracy, representativeness, completeness, comparability, and sensitivity. Data quality indicators include the following:

- Target analytes are measured in method blanks to help identify and eliminate potential contamination sources in a sample batch.
- LCS and LCSD recoveries are measured and RPDs between recoveries are calculated to determine if corrective action must be taken.
- RPDs are calculated between field duplicate and primary sample results to assess sources of variability arising from the field sampling protocol and distribution of target analytes within the sample matrix. Duplicate samples are submitted to the laboratory "blind" to assess the laboratory's precision.

- Trip blanks are analyzed to determine the magnitude and identify the likely source(s) of potential contamination problems resulting from lab and/or field activities, and to assess their contribution to measurement error.
- Surrogate recoveries are calculated as indicators of laboratory accuracy and data quality.

Data quality review reports and ADEC data quality checklists will be completed for each data package. The data quality review will be performed by the project chemist, and assisted by the laboratory project manager as necessary.

4.7 Plan for Obtaining the Data

Two samples will be collected from exposed asphalt tar on the beach and three pore water samples will be collected along the edge of the Indian River, from locations above the site, at the site and below the site.

(This Page Intentionally Left Blank)

5 Field Sampling Plan

The procedures outlined in this section will be followed for pore water and asphalt sampling in the field. The following SOPs will be used and are included in Appendix A:

- SOP01 – Logbook Documentation and Field Notes
- SOP10 – Quality Control Samples
- SOP11 – Sample Chain of Custody
- SOP12 – Labeling/Packaging/Shipping Samples
- SOP13 – Equipment Decontamination
- SOP34 – Pore Water Sampling

5.1 Pore Water Sampling

Three locations on the beach will be identified for pore water sampling prior to field mobilization (Figure 2). The 1/8-inch diameter, 14-inch long MHE® pore water sampler will be inserted 10 to 12 inches into the soil/sediment in no more than three inches of water (depending on wave action) at the water/shoreline interface. Single-use Teflon® hose will be attached to the sampler and, using a peristaltic pump, water will be pumped at a rate of approximately 50 mL per minute. A graduated cylinder will be used to measure the pumping rate. Initial water is expected to be sediment-laden. When the water clears, it will be pumped to remove a minimum of 35 additional mL of water (three probe volumes). Samples will then be collected for VOC and SVOC. One duplicate sample will be collected for each analysis. These samples will be labeled and shipped to the analytical laboratory in Tacoma, Washington to be analyzed for VOC and SVOC.

5.1.1 Pore Water Sampling Locations

Proposed pore water sample locations are displayed on Figure 3.

5.1.2 Pore Water Sampling Protocol

Pore water samples will be collected as described above in Section 5.1, in accordance with SOP34. QC samples will be collected according to SOP10. Samples will be labeled, packed, and shipped according to SOP12 and COCs will be filled out according to SOP11. All SOPs are included in Appendix A.

5.1.3 Pore Water Sampling Health and Safety

Health and safety concerns for pore water sampling include contact and inhalation of water containing VOCs and SVOCs. Mitigation procedures are included in the project-specific Health and Safety Plan in Appendix C.

5.1.4 Pore Water Field Measurements

Field screening is not necessary and will not be implemented for this project.

5.1.5 Pore Water Analytical Measurements/Methods

All pore water samples will be analyzed for VOCs by USEPA Method 8260C, and for SVOCs by USEPA Method 8270D-SIM. Table 5-1 presents the analytical methods and quality control program for the site characterization. Laboratory performance and analytical results will be checked through a quality assurance review, which will include ADEC's Laboratory Data Review Checklist. The review will assess analytical quality through six data quality indicators: precision, accuracy, representativeness, comparability, completeness, and sensitivity.

TABLE 5-1: PORE WATER ANALYTICAL METHODS AND QUALITY CONTROL PROGRAM

Parameter / Analytical Methods	Matrix	Number of Samples	Duplicates	Frequency of Trip Blanks	Holding Time
VOC / EPA 8260c	Water	3	1	1 per cooler	14 days
SVOC / EPA 8270D-SIM	Water	3	1	N/A	7 days

Key:
AK Alaska
EPA U.S. Environmental Protection Agency
N/A not applicable
VOC volatile organic compounds
SVOC semivolatile organic compounds

5.2 Asphalt Sampling

Two representative pieces of asphalt will be identified and collected from the beach on site (Figure 2). These samples will be labeled and shipped to the analytical laboratory in Tacoma, Washington to be extracted using SPLP and analyzed VOC and SVOC.

5.2.1 Asphalt Sampling Protocol

Asphalt samples will be collected as described above in Section 5.1. QC samples will be collected according to SOP10. Samples will be labeled, packed, and shipped according to SOP12 and COCs will be filled out according to SOP11. All SOPs are included in Appendix A.

5.2.2 Asphalt Sampling Health and Safety

Health and safety concerns for pore water sampling include contact and inhalation of water containing VOCs and SVOCs. Mitigation procedures are included in the Site-Specific Health and Safety Plan in Appendix C.

5.2.3 Asphalt Field Measurements

Field screening is not necessary and will not be implemented for this project.

5.2.4 Asphalt Analytical Measurements/Methods

All asphalt samples will be analyzed for VOCs by USEPA Method 8260C, for SVOCs by USEPA Method 8270D, and for SPLP by EPA Method 1312. Table 5-1 presents the analytical methods and quality control program for the site characterization. Laboratory performance and analytical results will be checked through a quality assurance review, which will include ADEC's Laboratory Data Review Checklist. The review will assess analytical quality through six data quality indicators: precision, accuracy, representativeness, comparability, completeness, and sensitivity.

TABLE 5-2: ASPHALT ANALYTICAL METHODS AND QUALITY CONTROL PROGRAM

Parameter / Analytical Methods	Matrix	Number of Samples	Duplicates	Frequency of Trip Blanks	Holding Time
SPLP / EPA 1312-VOC / EPA 8260C	Solid	2	1	1	14 days
SPLP / EPA 1312-SVOC / EPA 8270D	Solid	2	1	1	14 days

Key:
AK Alaska
EPA U.S. Environmental Protection Agency
N/A not applicable
SPLP synthetic precipitation leaching procedure
SVOC semivolatile organic compounds
VOC volatile organic compounds

5.3 Sample Handling

This section describes the sample handling protocol for environmental samples collected during the investigation.

5.3.1 Sample Designation

Each sample will receive a unique designator. Unique designators may be an alpha-numeric combination that signifies the location or decision area, matrix, depth or river reach, etc.

5.3.2 Sample Labeling

All samples will be labeled with the following:

- Sample ID
- Matrix
- Date and time collected

- Preservative (if applicable)

5.3.3 Sample Handling and Chain of Custody

- All samples will be placed in a cooler with sufficient gel ice to keep sample temperatures at 4 degrees Celsius (°C) ± 2°C until delivery to the project laboratory under standard COC procedures. A trip blank and a temperature blank will be included with each cooler.
- Sample coolers will be shipped to TestAmerica in Tacoma, Washington for analysis. The project manager is Elaine Walker.
- The laboratory-provided COC forms will be used to track the possession of each sample from the time it is collected to the time it is accepted by TestAmerica. COC procedures will be followed as outlined in SOP11, included in Appendix A. One COC will be required per cooler. An example COC form is provided along with other field forms in Appendix D.

5.3.4 Documentation and Records

A written record of all field activities will be kept in a field logbook. All entries will be legible, written in waterproof ink, and contain accurate and inclusive documentation of the field activities. Errors or changes will be noted using a single line to cross out the entry and will be dated and initialed. The logbook will be maintained as part of the permanent record for the site. All field logbook entries will be dated and signed. Activities and observations to be noted in the logbook include the following:

- Name of author and date and time of entry
- Documentation of equipment calibration
- Location of activity and site conditions
- Names and affiliations of onsite personnel
- Field observations and comments
- Weather conditions
- Rationale for sampling locations and for any changes to sampling protocol
- Locations of site photographs
- Site sketches with sample location measurements
- Health and safety comments

5.4 Investigative-Derived Waste Handling

Waste streams expected from this project are expected to be non-hazardous and include decontamination and purge water, and disposable PPE and sampling materials.

- Purge water generated during pore water sampling is expected to be less than one gallon. This water will be disposed of on the ground in an upland portion of the site unless a petroleum sheen or odor is noted. In that case, the water will be containerized and disposal options discussed with NPS and ADEC

- All other disposable sampling equipment and PPE will be bagged and disposed of in a location as directed by onsite NPS personnel.

5.5 Health and Safety

A Site-Specific Health and Safety Plan is included in Appendix C.

(This Page Intentionally Left Blank)

6 Data Management

Ahtna will maintain copies of all field forms, work plans, reports, and associated project materials. See Table 6-1 (next page) for a listing of each item and the team member responsible.

TestAmerica will consolidate all laboratory analyses (including sample and QC results) into a laboratory data report. The laboratory data report will be presented in both hard copy and electronic copy deliverables. The electronic data deliverables will be provided in the electronic data format. All documents will be checked for completeness prior to submittal.

All statements and data provided in TestAmerica Laboratory Data Reports shall be in conformance with provisions set forth by the TestAmerica Quality Assurance Program Plan, the National Environmental Laboratory Accreditation Conference, and this SAP, unless noted otherwise.

The laboratory report shall include, at a minimum, the following information:

- Case narrative
- COC records (client and internal)
- Sample receipt and login records
- Sample chronology
- Quantitation and detection limit verification
- Standards traceability
- Instrument calibration records
- Laboratory qualifier definitions
- Project sample results
- QC sample results
- Corrective action reports

Table 6-1 Data Management Table

Record	Generation	Verification	Storage Location/Archival
Sample Collection and Field Records			
Field Logbook	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Calibration Records	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
COC Records	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Shipping Records	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Daily QC Reports	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Corrective Action Reports	Ahtna Field Crew (Alexa FitzGerald)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Laboratory Records and Deliverables			
Narrative	TestAmerica PM (Elaine Walker)	Ahtna Chemist (Emily Freitas)	Electronic Project Folder
COC Records	TestAmerica PM (Elaine Walker)	Ahtna Chemist (Emily Freitas)	Electronic Project Folder
Summary Results	TestAmerica PM (Elaine Walker)	Ahtna Chemist (Emily Freitas)	Electronic Project Folder
QC Results	TestAmerica PM (Elaine Walker)	Ahtna Chemist (Emily Freitas)	Electronic Project Folder
Data Packages (PDF- Level III)	TestAmerica PM (Elaine Walker)	Ahtna Chemist (Emily Freitas)	Electronic Project Folder
Data Assessment Documents and Records			
Data Validation Report	Ahtna Chemist (Emily Freitas)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Data Usability Assessment	Ahtna Chemist (Emily Freitas)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Data Verification Checklists	Ahtna Chemist (Emily Freitas)	Ahtna PM (Nino Muniz)	Electronic Project Folder
Waste Documents			
Disposal Document	Waste Management	Ahtna PM (Nino Muniz)	Electronic Project Folder
Waste Manifest	Waste Management	Ahtna PM (Nino Muniz)	Electronic Project Folder

7 Assessment and Oversight

This section describes the measures that will be employed to ensure that this SAP is implemented properly.

7.1 Assessment and Corrective Actions

7.1.1 Field Audit and Response Actions

During field activities, QA audits will be completed to ensure that the procedures outlined in this SAP are being followed. Alexa FitzGerald is responsible for QA/QC. The samples collected for laboratory analysis will be entered onto COCs and cross-checked with the sample label to verify all sample names, dates, and times are correct. If discrepancies are found between the COC and samples, the QA/QC responsible person will make the necessary corrections and initial any changes made. The discrepancies will be noted in the field logbook for that day and reported to the project PM.

7.1.2 Laboratory Audit and Response Actions

The laboratory quality control manual and all QC procedures associated with each method can be found in Appendix B.

7.2 Quality Assessment Reporting

A QA report detailing any deviations from the SAP will be generated at the end of the project or as required. Project QC forms will be maintained in three-ring binders at the site and will be readily available. Other forms to be used on this project include but are not limited to the following:

- Copies of all contract modifications, arranged in numerical order, including documentation that modified work was accomplished
- An up-to-date copy of the deficiency tracking system
- Audit checklists (if necessary)

All field records will be maintained at the site until fieldwork is completed. At that time, the field team will transfer all records for archival in the project file. Reports will be generated by the Ahtna field team, and reviewed by the PM.

7.2.1 Data Verification

All field documents, along with laboratory deliverable, will be collected and verified. This step includes, but is not limited to, ensuring that data for all samples have been provided, all relevant laboratory internal QC data (including raw data) have been provided in the report, and the specified analytical methods were used by the laboratory.

7.2.2 Data Validation

Ahtna will perform a validation assessment of data quality on all laboratory data. This validation step includes, but is not limited to, documenting the data verification process, summarizing the samples and analyses, reviewing samples and analyses, reviewing sample handling, reviewing the laboratory QC data, assigning qualifiers, reviewing the QA data from the third party laboratory, reviewing LOQ and LODs to determine if non-detect results are greater than the project quantitation limits (PQL), defining LOQs greater than the PQLs, compiling a table of rejected data, and compiling a sample summary table.

7.3 Reconciliation with DQOs and Data Usability

Ahtna will use the Data Assessment Goals defined in Table 4-4 as guidance for data quality objectives. Deviations from these goals and laboratory DQOs will be evaluated against the data collected to determine usability.

8 Investigation Outputs

A final SI report will be completed to meet the NPS requirements, as stipulated in the scope of work. The report will be prepared in pre-draft, draft and final form with responses to the NPS and/or ADEC's comments addressed in a written response to comments. The report shall utilize the NPS SI report format and include the following information:

- Cover page for the SI Report with the name and signature of the NPS PM and SITK Park representative
- A narrative report describing fieldwork activities, including variances from the planning documents
- Tables, drawings, and figures to support the narrative report, summarize site data, show locations of field activities, and illustrate processes and decision matrices
- Appendices containing copies of all chemical data generated; copies of all permits; copies of waste manifests, waste profile sheets, certificates of disposal, and other pertinent documentation; copies of all field notes, logs, forms, and Daily Contractor QC Report, and photographs
- Analytical data review, which summarizes the completed ADEC Laboratory Data Review Checklists; these checklists shall be complete and submitted with laboratory data in the draft report
- Recommendations for the site.

(This Page Intentionally Left Blank)

9 References

Alaska Administrative Code (AAC) Title 18 Chapter 75 (18 AAC 75). Revised as of January 1, 2016.

City and Borough of Sitka Public Works Department Environmental Division (City and Borough of Sitka). 2015. Drinking Water Quality Report for the Year 2015, Sika, Alaska. (Public Water System AK2 130075). http://www.cityofsitka.com/government/departments/publicworks/documents/DrinkingWaterQualityReport2015.pdf. Accessed May 30, 2018.

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 United States Code Section 9601.

Intergovernmental Data Quality Task Force (IDQTF). 2005. Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. EPA/505/B-04/900A; DoD DTIC ADA 427785.

Interstate Technology Regulatory Council (ITRC). 2012. Technical and Regulatory Guidance: Incremental Sampling Methodology. February.

National Park Service (NPS). ND. *Natural Resource Management Reference Manual #77.* http://www.nature.nps.gov/rm77/. Accessed February 2, 2014.

National Park Service (NPS). 2018. Statement of Work: Indian River Asphalt Plant Preliminary Assessment/Site Inspection, Sitka National Historic Park. January 12.

NPS. 2016. Hydraulic Activity, Sitka National Historic Park, Alaska. https://www.nps.gov/sitk/learn/nature/hydrologicactivity.htm. Last updated April 22, 2016. Accessed May 30, 2018.

NPS. 2015. Geologic Formations, Sitka National Historic Park, Alaska. https://www.nps.gov/sitk/learn/nature/geologicformations.htm. Last updated April 14, 2015. Accessed May 30, 2018.

NPS. 2014b. Conceptual Site Model Guidance.

NPS. 2014c. NPS Protocol for the Selection and Use of Ecological Screening Values for Non-Radiological Analytes. Revision 1. January 13.

NPS. 2014d. NPS Data Quality Primer.

Shannon & Wilson, Inc. (Shannon&Wilson). 1995. Environmental Site Assessment, Indian River Asphalt Site, Sitka National Historic Park, Sitka, Alaska. April 7.

United States Climate Data. 2018. Climate for Sitka, Alaska. https://www.usclimatedata.com/climate/sitka/alaska/united-states/usak0224. Accessed June, 2018.

United States Environmental Protection Agency (USEPA). USEPA Region 9, Regional Screening Levels (RSLs). http://www.epa.gov/region9/superfund/prg/. Accessed September 22, 2014.

USEPA. 2012. Guidance for Evaluating and Documenting the Quality of Existing Scientific and Technical Information. Addendum to: *A Summary of General Assessment Factors for Evaluating the Quality of Scientific and Technical Information*. December.

USEPA. 2006a. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA/240/B-06/001. February.

USEPA. 2006b. Data Quality Assessment: Statistical Methods for Practitioners. EPA/240/B-06/003. February.

USEPA. 2006c. Data Quality Assessment: A Reviewer's Guide. EPA/240/B-02/002. February.

USEPA. 2003a. A Summary of General Assessment Factors for Evaluating the Quality of Scientific and Technical Information. EPA/100/B-03/001. June.

USEPA. 2003b. Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples. EPA/600/R-03/027. November.

USEPA. 2002a. Guidance for Quality Assurance Project Plans. EPA/240/R-02/009. December.

USEPA. 2002b. Guidance on Choosing a Sampling Design for Environmental Data Collection: for Use in Developing a Quality Assurance Project Plan. EPA/240/R-02/005. December.

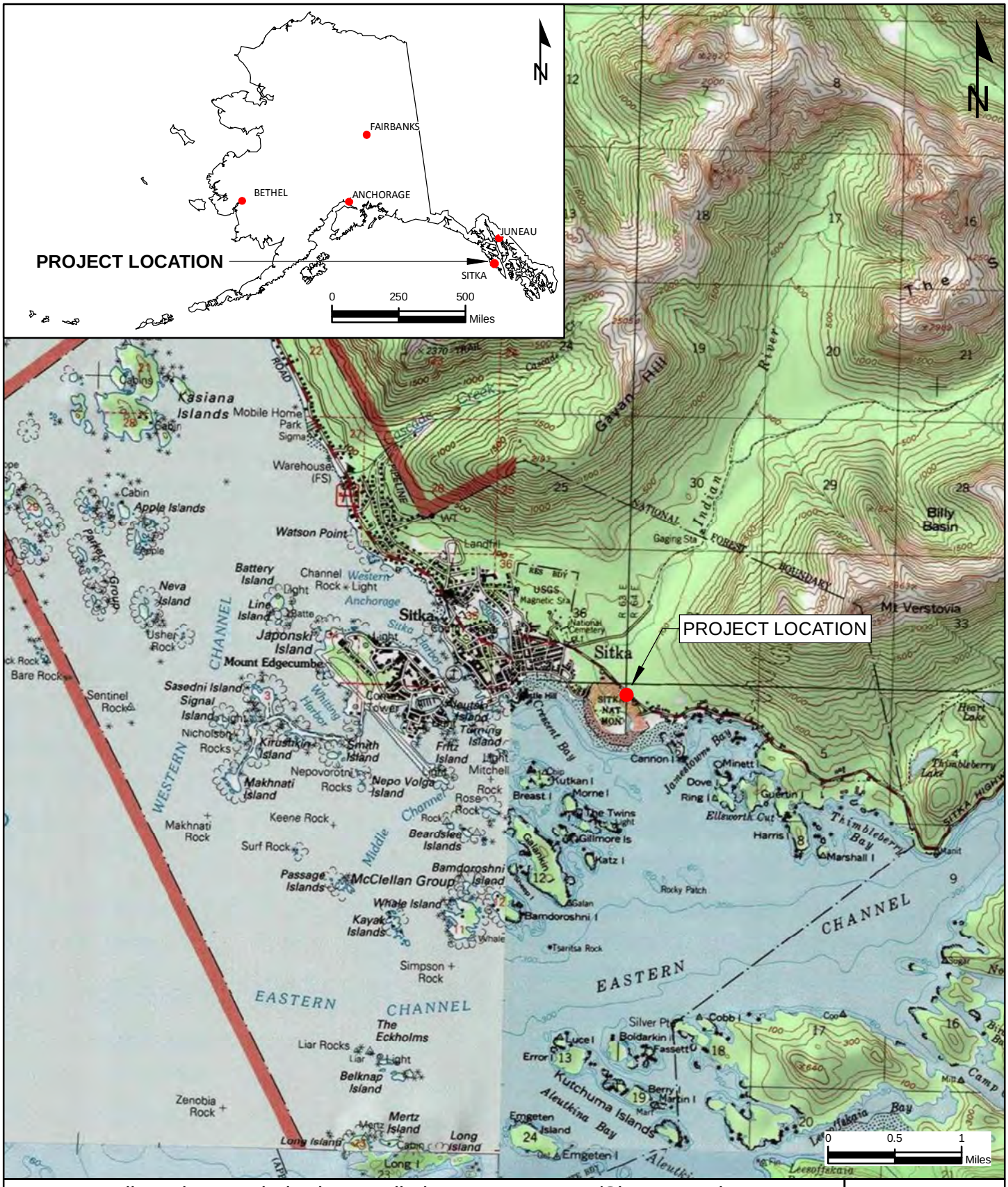
USEPA. 2002c. Guidance on Environmental Data Verification and Data Validation. EPA/240/R-02/004. November.

USEPA. 2001. EPA Requirements for Quality Assurance Project Plans. EPA/240/B-01/003. March.

USEPA. 1992. Guide to Management of Investigative-Derived Wastes. Publication: 9345.3-03FS. January.

US Geological Survey (USGS). 2008. Techniques of Water-Resources Investigations: National Field Manual for the Collection of Water-Quality Data.

USGS. 2002. Techniques of Water-Resources Investigations of the United States Geological Survey: Statistical Methods in Water Resources. Book 4, Hydrologic Analysis and Interpretation. September.



Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

Ahtna Engineering Services, LLC

State & Site Vicinity Map

Project Number: 20279.011	Figure Number: 1
Date: 6/7/2018	
Drafted By: R.F.	

Prepared by rfrye, 6/7/2018: C:\Users\frye\OneDrive - Ahtna, Inc\Desktop\GIS\Sitka\GIS\MXD\F1_State&SiteVicinity.mxd

(This Page Intentionally Left Blank)

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

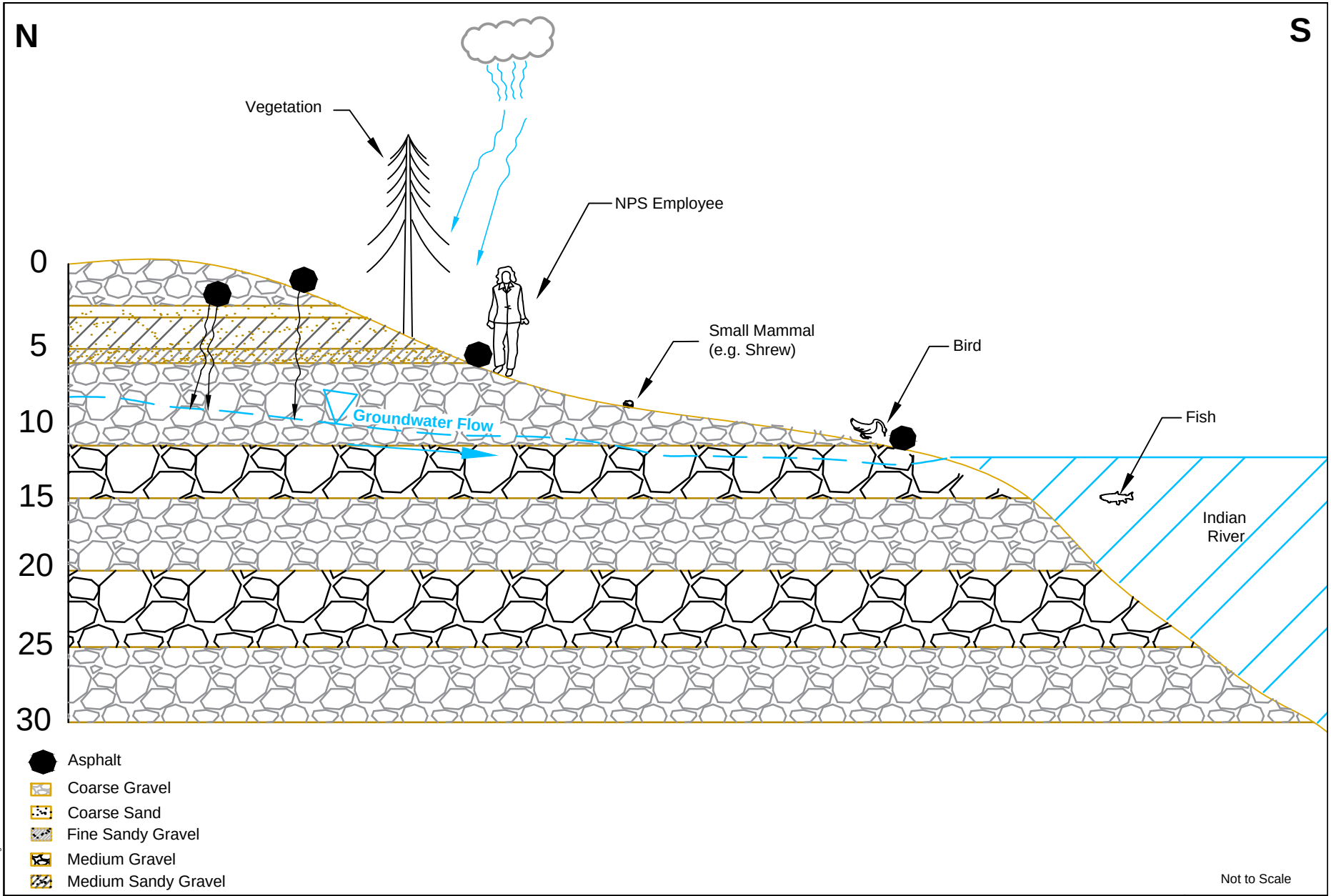
Ahtna Engineering Services, LLC

Site Map and Proposed Pore Water Sample Locations

Project Number:	Figure Number:
20279.011	2
Date: 8/22/2018	
Drafted By: R.F.	

Prepared by rfrye, 8/22/2018; C:\Users\rfrye\OneDrive - Ahtna, Inc\Desktop\GIS\Sitka\GIS\MXD\F2_AreasOfConcern.mxd

(This Page Intentionally Left Blank)



Not to Scale

Indian River Asphalt Plant Preliminary Assessment/Site Inspection
National Park Service
Sitka National Historic Park, Alaska

Ahtna Engineering Services, LLC

Conceptual Site Model

Project Number:	Figure Number:
20279.011	3
Date: 06.21.2018	
Drawn By: R.F.	

C:\Users\rfrye\Desktop\Sitka\CAD\F-4ConceptualSiteModel.dwg

(This Page Intentionally Left Blank)

Appendix A – Standard Operating Procedures

(This Page Intentionally Left Blank)

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE
FOR LOGBOOK DOCUMENTATION AND FIELD NOTES
No. 01

1.0 INTRODUCTION

1.1 Purpose

The purpose of this standard operating procedure (SOP) is to direct field personnel in the techniques and requirements for recording information in logbooks and to ensure that field activities are properly documented.

Adequate documentation is necessary to describe the work performed. Attention to detail is vital as field logbooks have been shown to be useful in administrative and judicial proceedings and for cost recovery measures.

1.2 Scope

The scope of this SOP is to describe the data entry requirements and suggested format for field log books.

2.0 RESPONSIBILITIES

Field Personnel – Each person in the field is responsible for maintaining a field logbook.

Project Manager (PM) – The PM is responsible for reviewing the adequacy of the logbooks during and after fieldwork.

3.0 DEFINITIONS

Field logbook – A bound, weatherproof notebook used to record daily field activities and act as a historical factual record of events. Field logbooks are permanently assigned to a specific project.

Field datasheets – Any documentation that is supportive of the field logbook information that is important for preserving an accurate historic record of field activities but is recorded on unbound paper. These records should be referenced in the field logbook and include groundwater sampling datasheets, equipment calibration datasheets, photograph logs, soil boring logs, chain of custody forms, shipping manifests, daily tailgate meeting records, etc.

Electronic Datasheets – Any documentation that is supportive of the field logbook information that is important for preserving an accurate historic record of field activities but is recorded

electronically through field instruments. These records should be referenced in the field logbook and include global position system (GPS) coordinates, pressure transducer data, photographs, etc.

4.0 EQUIPMENT

The following equipment is required:

- A bound field logbook with pre-numbered consecutive pages
- Waterproof, indelible pens/markers

5.0 PROCEDURE

5.1 Field Logbooks

Each logbook shall contain the following information on the cover:

- Owner of the book
- Book number
- Job name and work order
- Start date
- End date

It is useful to include project contact information on the inside front cover or first page of the logbook. Contact information includes names and phone numbers of subcontractors, project assistants, field team members, and emergency numbers from the site-specific health and safety plans.

Each logbook page shall include the following:

- Job name or number and date at the top of each page
- Date and signature at the bottom of each page, over any remaining blank lines

Logbooks entries shall adhere to the following guidelines:

- Pages shall never be removed from the logbook
- All information must be printed legibly and in waterproof, indelible ink
- Entries shall be written using objective and factual language and should be made in chronological order
- Entries shall be made on subsequent lines such that no blank lines exist on each page
- If any space remains on the bottom of the last page of field entries at the conclusion of the day's entries, a diagonal line shall be drawn to obscure any additional entries on that page
- If corrections are necessary, a single line may be drawn through the original entry. The corrected information may then be added and should be initialized and dated.

At a minimum, the standard daily entries shall include the following:

- Project name and location
- Page number
- Date and time; time shall be based on the 24-hour clock (i.e., 2100 instead of 9 pm)
- Weather conditions and changing weather that may impact site conditions
- Site conditions and other salient observations
- Full names and titles/roles of personnel on-site, including visitors
- Daily objectives
- Time and location of activities
- Work start/stop times
- Level of PPE
- All relevant field observations, major task decisions, comments, or other valuable site investigation information
- References to relevant datasheets and documentation preserved outside the logbook such as groundwater sampling datasheets, soil boring logs, etc. Do not duplicate information from the referenced sheets in the logbook.
- Location of work areas (sketches or photographs when appropriate, with north arrow and approximate scale)
- Survey and/or location of any sampling points, including swing-tie measurements
- Type of field instrumentation (model number and serial number) and all calibrations performed
- Decontamination times and methods
- All field measurements
- Type, amount, and method of disposal for investigation-derived waste
- Changes/deviations from the work plan and reason for deviations
- Any general observations or notes
- Daily equipment calibration and maintenance
- Sample record (sample identification, date, time, media, number of samples, and location)

Correct erroneous field record or log book entries with a single line through the error. Do not erase incorrect information. Date and initial revised entries. Logbooks and field forms will be kept in the project file when complete or when not in use.

5.2 Field Datasheets

All unbound data documentation are part of the field records and should be maintained with safe document handling and archiving procedures. These records should be recorded in waterproof, indelible ink and on weatherproof paper as necessary. As soon as possible, the unbound records shall be scanned to create an electronic record to ensure document preservation.

5.3 Electronic Datasheets

All electronic data that are part of the field records shall be downloaded to a designated location and maintained for project use. Care must be taken when downloading the electronic data to ensure

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE
FOR QUALITY CONTROL SAMPLES
No. 10

1.0 INTRODUCTION

1.1 Purpose

The purpose of this standard operating procedure (SOP) is to direct field personnel in the techniques and requirements for collecting field quality control (QC) samples from any matrix. Field QC samples are collected to ensure the reliability and validity of field and laboratory data.

1.2 Scope

The scope of this SOP is to describe the purpose and methods for collection of QC samples by Ahtna personnel for all sample matrices. The types and quantities of QC samples will be determined per project in the site-specific work plans.

2.0 RESPONSIBILITIES

Project Manager (PM) – The PM is responsible for providing adequate resources to the field staff and ensuring that field staff has adequate experience and training to successfully comply with the SOP. The PM is responsible for approving and documenting techniques that are not specifically described in this SOP but are considered the best QC methods for the current project. The PM is responsible for ensuring that project plans are complete and reviewed and approved by the appropriate personnel and organizations.

Site Supervisor (SS) – The SS is responsible for coordination of field activities including adhering to site-specific plans and ensuring that personnel are properly trained in the techniques necessary to follow this SOP.

Quality Control Manager – The QC manager is responsible for designing a QC plan and ensuring that the field staff has an understanding of the methods and procedures to implement the QC plan.

Sampler – The sampler is responsible for the collection of QC samples as specified in this SOP.

3.0 DEFINITIONS

Aliquot - A portion of a sample.

Ambient blank – A blank sample of reagent-grade water poured into a volatile organic compound (VOC) sample vial at the sampling site near other VOC sample collection. Used to assess the introduction of contaminants from ambient sources such as fuel motors in operation.

Background sample – A sample collected from an area similar to the one being sampled but located in an area free of contaminants.

Data quality objectives – Quantitative and qualitative statements that clarify the study objectives, define the most appropriate type of data to collect, determine the appropriate conditions from which to collect the data, and specify tolerable limits on decision errors that will be used as the basis for establishing the quality and quality of data needed to support site decisions.

Equipment blank – A blank sample of analyte-free water, typically supplied by the laboratory, poured into, through, or over equipment used for sampling and collected in a sample container. Used to assess the efficacy of decontamination procedures and therefore should be collected immediately following equipment decontamination. Per project specification, a minimum of one equipment blank will be collected per 20 samples per matrix.

Field Blank – A sample of deionized water or preservative poured into a sample container in the field, and shipped to the laboratory with field samples. Per project specification, a minimum of one field blank will be collected per 20 samples per matrix.

Field duplicate – Two samples taken from, and representative of, a single location and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are homogenized prior to placing the matrix into a sample container. Used to assess variance of the total method including sampling and analysis.

Field replicate – Two samples taken from, and representative of, a single location and carried through all steps of the sampling and analytical procedures in an identical manner. Replicate samples are not homogenized prior to placing the matrix into a sample container but are collected in rapid succession. Used to assess variance of the total method including sampling and analysis.

Matrix spike / matrix spike duplicate – An aliquot of a client sample spiked in the laboratory with known concentrations of all analytes listed in the method at a level less than or equal to the midpoint of the analytical calibration curve for each analyte. Used to document the accuracy and bias of a method due to the sample matrix and therefore should be collected from a sample area free of contaminants, if possible.

Quality control samples – Samples used to check the operation of a measurement system to obtain a measure of the quality of data generated.

Quality assurance project plan (QAPP) – A specific plan for the collection of data used to assess the quality of data generated for a project.

Relative percent different (RPD) – A measure used to evaluate the difference between contaminant concentrations in two samples. This is primarily used for duplicate samples. The equation is as follows: $RPD = \frac{X_1 - X_2}{(X_1 + X_2)/2} \times 100$

Temperature blank – A blank sample of water, typically provided by the laboratory, and placed in sample coolers before or during sample collection to ensure temperature equilibration with samples. Used to determine the temperature at which samples were delivered to the laboratory for analysis and required for samples needing temperature preservation. One per cooler.

Trip blank – A blank sample of preservative provided by the laboratory that accompanies volatile sample jars through their life-cycle. Used to assess the introduction of contaminants from sample containers or during transportation and storage procedures and for this reason trip blanks are not opened. Minimum of one per cooler.

4.0 EQUIPMENT

The equipment used for the associated sampling (SOP No. 02, 03, 04, 05) should be used for the collection of the QC samples. It is important that the sample collection procedures used to collect the primary samples be used for the collection of all QC samples. If preparing blanks in the field, reagent-grade water will be necessary.

5.0 PROCEDURE

The site-specific work plan will determine which QC sample types are required for the project. For the collection of the QC samples, follow the applicable procedures outlined in the following sections.

5.1 Field Duplicates and Field Replicates

The QC check of the field duplicate is a low relative percent difference (RPD) between the laboratory results for the primary and duplicate sample. An exceedance of the allowable tolerance limits suggests that the precision of the sampling effort is insufficient. Inadequate precision could be due to various issues including poor sampling methodology.

A minimum of one field duplicate must be collected for every 10 field samples for each matrix samples and for each target analyte. Field duplicates must be collected from locations of known or suspected contamination, and duplicate soil and water samples must be collected in the same manner and at the same time and location as the primary sample. For a sampling event occurring over multiple days, all field duplicates must not be collected in one day and the goal should be to collect a minimum of one field duplicate per day.

Field duplicates must be:

- Submitted as blind samples to the approved laboratory for analysis,
- Given unique sample numbers (or names) and sample collection times, and
- Adequately documented in the field records or log book.

To collect a field duplicate of a pumped water sample, ensure that the water being collected is representative of the field conditions and fill the laboratory-supplied jars in immediate succession for each analysis (replicate). For example, if three vials are required for the primary sample, collect six vials in succession and label three for the primary sample and three for the duplicate sample.

To collect a field duplicate of a non-volatile soil sample, determine the sample interval that ensures enough soil volume required to fill all of the jars. Gather and homogenize the soil and fill the two sample containers simultaneously. Preserve as required.

To collect a field replicate of a volatile soil sample, for each grab of soil, fill the primary and duplicate sample containers simultaneously, placing equal amounts of the soil in the jar for the primary and the jar for the duplicate. Preserve as required.

Field duplicates require disguised sample identification to the laboratory including a unique sample name and time.

5.2 Matrix Spike and Matrix Spike Duplicates

The purpose of a matrix spike (MS) sample is to evaluate matrix effects on the analysis method. The matrix spike duplicate (MSD) sample is used to corroborate the contaminant concentrations in the matrix spike sample, as measured by the RPD. At a location designated in the site-specific plan, and preferably from an area with little to no contamination, collect three separate samples from the same location at the same time. The samples should have the same matrix to ensure a valid result; if the sample interval does not consist of similar visual and olfactory observations, choose another location for collection of MS and MSD samples.

MS and MSD samples should be labeled with the same sample name and time as the primary sample and denoted on the chain of custody. The laboratory will analyze the parent sample to determine the background analyte concentrations present in the sample. The laboratory will then spike the MS and the MSD samples with known concentrations of analytes prior to analysis and run the analysis in the same manner as the parent sample. The background concentration from the parent sample will be subtracted from the MS and MSD results and the RPD calculated.

5.3 Blanks

The primary purpose if quality control blanks (i.e. trip, field, and equipment blanks) is to trace sources of artificially introduced contamination.

5.3.1 Field Blanks

Field blanks area a sample of preservative or deionized water poured into the laboratory-provided container in the field, and shipped to the laboratory with the field samples. Per project specifications, a minimum of one field blank will be collected per 20 samples per matrix and per analyses.

5.3.2 Equipment Blank / Rinsate Blank

The purpose of an equipment blank is to evaluate the efficacy of a decontamination procedure of non-disposable sampling equipment. Equipment blanks are samples if analyte-free water poured over or through decontaminated field sampling equipment prior to the collected of environmental samples. Per project specifications, a minimum of equipment blank will be collected per 20 samples per matrix and per analyses.

5.3.3 Temperature Blank

The purpose of a temperature blank is to record the temperature of all samples upon receipt at the laboratory. A temperature blank must be kept in the cooler with the samples at all times. If a temperature blank is not included in the cooler from the laboratory, one can be prepared in the field by filling a jar or bottle with tap water and labeling it as the temperature blank. Upon receipt by the laboratory, the temperature will be recorded on the chain of custody. Samples and temperature blanks must be kept under 6 °C.

5.3.4 Trip Blank

Trip blanks area a clean sample of a matrix that is taken from the laboratory to the site and then transported back to the laboratory without having been exposed to the sampling procedures. The purpose of a trip blank is to assess the introduction of contaminants from sample containers or during transportation and storage procedures. Trip blanks are required for volatile analyses and must accompany all volatile sample jars. For this reason, it is best to put all volatile analysis jars into one cooler to allow for a limited number of trip blanks, as one trip blank is required per cooler of volatile samples. Exceeding allowable tolerance limits for trip blanks suggests that contamination was introduced during shipping and field handling procedures.

5.4 Labeling

All quality control samples should be labeled and included on the chain of custody, with the exception of the temperature blank. The naming conventions should be specified in the site-specific work plan. In the absence of a naming convention, adding a suffix of EB for equipment blank and TB for trip blank should suffice.

6.0 REFERENCES

Environmental Protection Agency (USEPA), 1990. *Samplers Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December.

Alaska Department of Environmental Conservation (ADEC), 2017. *Field Sampling Guidance*, August.

7.0 REVISION LOG

Revision Date	Author	Revision Details
12/26/13	Olga Stewart	Initial Issue

4/7/16	Andrew Weller	Minor Grammar Edits
11/30/16	Ashley Olson	Updated based in the new ADEC sampling guidance, updated logo
9/1/2017	Lexie Lucassen	Updated text based on August 2017 ADEC *Field Sampling Guidance*; updated reference

that the original record is preserved. Naming conventions should be used to indicate the project, date, and other relevant information to ensure accurate use.

5.4 Document Control

At the conclusion of a task or project, all field documentation, including the field logbook, field datasheets, and electronic data, should be submitted to the project manager for records retention. All original documents should be kept in the project file. Copies of all field notes and field records will be included in reports.

6.0 REFERENCES

Alaska Department of Environmental Conservation (ADEC), 2017. *Field Sampling Guidance*, August.

ADEC, 2017. *Site Characterization Work Plan and Reporting Guidance for Investigation of Contaminated Sites*, March.

7.0 REVISION LOG

Revision Date	Author	Revision Details
1/2/14	Brandie Hofmeister	Initial Issue
4/15/16	Andrew Weller	Reference Section: ADEC Field Sampling Guidance Updated, Project-Specific Statement of Work Removed
12/1/2016	Ashley Olson	Logo
2/23/17	Katelyn Barnett Decker	Logo
9/1/17	Lexie Lucassen	Updated ADEC *Site Characterization Work Plan and Reporting Guidance* and *Field Sampling Guidance* references and incorporated into text

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE FOR SAMPLE CHAIN OF CUSTODY (COC) No. 11

1.0 INTRODUCTION

1.1 Purpose

The purpose of this standard operating procedure (SOP) is to direct field personnel in the techniques and requirements for maintaining the sample chain of custody (COC).

Proper handling, chain of custody, and documentation are necessary to provide an accurate written record to track the possession, handling, and location of samples from the moment of collection through reporting.

1.2 Scope

The scope of this SOP is to cover aspects of sample handling, with respect to custody, and the proper techniques for documenting the custody on the COC form.

2.0 RESPONSIBILITIES

Project Manager (PM) – The PM is responsible for providing adequate resources to the field staff and ensuring that field staff has adequate experience and training to successfully comply with the SOP. The PM is responsible for approving and documenting techniques that are not specifically described in this SOP but are considered the best sampling methods for the current project.

Sampler – The sampler is responsible for the handling and documentation of sample custody as specified in this SOP.

3.0 DEFINITIONS

Chain of custody (COC) – The chronological documentation of sample custody, showing the control, transfer, and analysis of samples.

Custody seal – An adhesive label placed across an opening that is used to detect tampering with samples after they have been packed for shipping.

Sample – A material that is housed in containers and identified with a unique sample identification number that is to be analyzed by a laboratory.

Sample custody – A sample is considered under custody if it is in your possession, if it is in your view after having been in your possession, if it was in your possession and is then locked up to prevent tampering, or if it is in a designated and identified secure area.

Sample label – An adhesive paper or tag that is placed on sample containers to designate a sample identification number and other identifying information.

4.0 EQUIPMENT

Equipment needed for chain of custody documentation includes the following:

- Sample jars that have been filled and labeled in accordance with the work plan
- Quality control (QC) sample containers
- Coolers with return address written on inside lid
- COC forms
- Custody seals
- Gallon-sized re-sealable plastic bag
- Clear tape

Note that this SOP is intended to be used in conjunction with the following SOPs, and as such, the equipment and materials needed for those activities are not included in this SOP:

- Logbook Documentation and Field Notes (No. 01)
- Labeling, Packaging, and Shipping Samples (No. 12)

5.0 PROCEDURE

Sample identification documents will be carefully prepared so that sample identification and chain of custody are maintained. Sample identification documents include the field logbook, sample labels, custody seals, and COC records.

A sample is in custody if it meets one of the following conditions:

- In an authorized person's physical possession
- In an authorized person's view after being in possession
- Was in an authorized person's possession then locked up
- Kept in a secured area that is restricted to authorized personnel

5.1 Field Custody Procedures

The following procedures shall be used by field personnel:

- As few persons as possible will handle samples.
- The sample collector will be personally responsible for the care and custody of samples collected until they are transferred to the laboratory.
- The sample collector will record sample data (time of collection, sample number, analytical requirements, and matrix) in the field logbook.

- Sample labels shall be completed for each sample, using waterproof ink.

5.2 Chain of Custody Record

All samples will be accompanied by a COC record. The COC form is typically provided by the laboratory unless otherwise specified in the work plan. The chain of custody record will be fully completed in duplicate. Information to be included on a chain of custody form includes the following:

- Project name and number
- Contractor name and address
- Laboratory name and address
- Name of person that collected the sample(s)
- Sample identification number
- Sample date and time (time in 24-hour format)
- Laboratory analysis methods required for each sample jar
- Preservatives added to each sample jar
- Sample matrix (soil, water, or other)
- Number of containers per sample
- Airway bill tracking number (if applicable)

Additional remarks can be added to the COC record to alert the laboratory including the following:

- Matrix spike/matrix spike duplicate (MS/MSD) sample volume. The note "MS/MSD" should be added within the same line as the primary sample.
- A request for rapid turnaround time.
- A note regarding the potential concentrations in a highly contaminated soil sample.

Indication of a duplicate sample should never be included on a COC record.

5.3 Sample Packaging

Samples will be labeled and packaged according to the labeling, packaging, and shipping SOP (SOP 12). The COC record will accompany all sample shipments. One COC record shall be prepared for each shipment. One COC record will be prepared for each cooler, even if multiple coolers are included in one shipment. The cooler name and NPDLWO# are required on the COC. The samples in the cooler must be listed on the COC record.

The COC record will be placed in a re-sealable plastic bag, the bag sealed shut to prevent water intrusion from the ice in the cooler, and the bag taped to the inside lid of the cooler. If one sample is contained in two coolers (i.e. one sample has too many containers to fit in one cooler), then a copy of the COC record will suffice to accompany the second cooler as long as the original is in the first cooler and the copy is denoted as a copy.

The duplicate copy of the COC record will be retained by the sampler and distributed as necessary to the sample coordinators. Airway bills will also be retained with the COC record as documentation of transport.

Custody seals are pre-printed, adhesive-backed seals with security slots designed to break if the seals are disturbed. Seals will be signed and dated at the time of use. Sample shipping containers will be sealed in as many places as necessary to ensure that the container cannot be opened without tearing the custody seals. Typically one custody seal will be placed along the front opening, and one along the side opening of a cooler. Strapping tape will be placed over the seals to ensure that seals are not accidentally broken during shipment.

If a sampler hand transports the samples to the laboratory without sample shipment, custody seals are not required.

5.4 Transfer of Custody

When transferring the possession of samples from the field sampler to a transporter or to the laboratory, the sampler will sign, date, and note the time as "relinquished by" on the COC record. The receiver will also sign, date, and note the time as "received by" on the COC record. The date and time of the receiver and relinquisher shall be the same.

When samples are transported by a common commercial carrier such as Alaska Airlines or Federal Express, the carrier will not sign the COC record. However, the airway bill tracking number should be recorded on the COC record. For this reason, the date and time of the receiver and relinquisher will not match when shipping through a common commercial carrier.

5.5 Laboratory Custody Procedures

A designated sample custodian will accept custody of the shipped samples and verify that the sample identification number matches the COC record. Pertinent information about shipment, pickup, and courier will be entered in the "Remarks" section. Temperature of the coolers at the time of receiving will be noted on the COC record.

6.0 REFERENCES

Alaska Department of Environmental Conservation (ADEC), 2017. *Field Sampling Guidance*, March.

American Society for Testing and Materials (ASTM), 2010. *Standard Guidance for Chain of Custody Procedures*, ASTM D4840-99.

7.0 REVISION LOG

Revision Date	Author	Revision Details
1/2/14	Brandie Hofmeister	Initial Issue
6/2/16	Andrew Weller	Referenced 2016 ADEC Field Sampling Guidance and Minor Grammar Edits
12/1/2016	Ashley Olson	Updated logo
9/1/2017	Lexie Lucassen	Updated to August 2017 ADEC *Field Sampling Guidance*

(This Page Intentionally Left Blank)

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE
FOR LABELING/PACKAGING/SHIPPING SAMPLES
No. 12

1.0 INTRODUCTION

1.1 Purpose

The purpose of this standard operating procedure (SOP) is to direct field personnel in the techniques and requirements for labeling, packaging, and shipping samples.

1.2 Scope

The scope of this SOP is to cover all aspects of labeling samples for identification, packaging samples for safe transport, and shipping samples from the field to the laboratory for analysis, as conducted by Ahtna personnel.

2.0 RESPONSIBILITIES

Project Manager (PM) – The PM is responsible for providing adequate resources to the field staff and ensuring that field staff has adequate experience and training to successfully comply with the SOP. The PM is responsible for approving and documenting techniques that are not specifically described in this SOP but are considered the best methods for the current project.

Sampler/Technician – The sampler/technician is responsible for the collection and labeling of samples as specified in this SOP. The sampler/technician is responsible for ensuring adequate packaging and proper shipping as specified by this SOP.

3.0 DEFINITIONS

Air waybill - The shipping document that identifies the sender and addressee, transport carrier, size, and priority of a shipment transported by aircraft.

Dangerous goods or hazardous materials: A substance or material, including a hazardous substance, that the U.S. Department of Transportation (DOT) has determined can pose an unreasonable risk to health, safety, and property when transported in commerce and that DOT has designated as a hazardous material.

Environmental sample – Any sample that has less than reportable quantities of any hazardous constituents according to the Department of Transportation (DOT) 49 CFR Section 172.

Sample label – An adhesive paper that is placed on sample containers (soil, water) or a tag that is tied to a sample container (air) to designate a sample identification number and other identifying information.

4.0 EQUIPMENT

Equipment needed for labeling, packaging, and shipping samples includes:

- Coolers
- Heavy-duty plastic bags
- Plastic zip-top bags, small and large
- Clear tape
- Strapping tape
- Duct tape
- Bubble wrap and/or foam inserts
- Gel ice packs
- Custody seals
- Completed chain of custody (COC) record
- Completed Bill of Lading
- Labels ("Keep cool/refrigerate", "This end up", "Do not freeze", "Fragile", "Address", "Dangerous goods", "Excepted quantities", "Saturday delivery" (as necessary), etc.

Note that this SOP is intended to be used in conjunction with the following SOPs, and as such, the equipment and materials needed for those activities are not included in this SOP:

- Logbook Documentation and Field Notes (No. 01)
- Sample Chain of Custody (No. 11)

5.0 PROCEDURE

5.1 Labeling

Samples should be labeled using nomenclature defined in the applicable work plan. All sample labels should be written in indelible ink and contain the following information:

- Sample name/identification
- Date/time (in 24-hour format)
- Sampler's initials
- Analysis requested
- Job name/number
- Preservative

Adhesive sample labels should be placed directly on the sample containers. If the labels are not adequately adhered due to moisture, secure the label by placing clear packaging tape over the label. Sample containers that are weighed by the laboratory prior to use should not have any additional labels placed on the container as it affects the weight. For those containers, use the

label that is already provided on the jar. Only one label should be placed on each sample container.

5.2 Packaging

The following steps must be followed when packing sample containers for shipment:

1. Choose a cooler with structural integrity that will withstand shipment. Secure and tape the drain plug with duct tape.
2. Be sure that the caps on all containers are tight and will not leak. Make sure not to over tighten and break the cap.
3. Check to make sure that the sample labels are intact, completed with the correct information, that identification exactly matches the COC record.
4. Use sufficient ice in packaging to ensure that samples are received by the laboratory at the proper temperature of 4 °C ± 2 °C.
5. Wrap and package containers sufficiently to prevent cross-contamination and ensure that containers remain intact during shipment.

To ship samples with gel ice packs, follow the steps below. Note that gel ice for sample shipping should be laid flat prior to freezing for use when frozen. Partially melted or soft gel ice packs should not be used to pack coolers for transport. A minimum of 8 frozen gel ice packs are required to maintain sample temperature during transit for 24 hours.

1. Place a layer of frozen gel ice packs, lying flat on their sides, along the bottom of the cooler. Cover the ice packs with a layer of bubble wrap and then place a sorbent pad over the bubble wrap.
2. Place all sample containers in bubble wrap, bubble bags, in their original boxes, or in re-sealable bags with sorbent pads, depending on the type of container. One-liter glass bottles should be double-bagged to prevent damage during transport.
1. Place the containers into the cooler with caps up. No containers should be placed on their sides, as there is significantly less chance of breakage when packed vertically.
2. Place additional gel ice packs in between sample containers in a manner that maximizes surface contact with the containers. If packaging water samples, each sample container should be in contact with a gel ice pack.
3. Fill excess space between sample containers with additional bubble wrap.
4. Place another layer of bubble wrap along the top of the cooler, and as possible, place a layer of gel ice packs, lying flat on their sides, along the top of the cooler.
5. Fill remaining headspace with additional packing material.

5.3 Shipping

Environmental samples are shipped as non-hazardous material unless the samples meet the established DOT criteria for a “hazardous material” or the International Air Transport Association (IATA)/International Civil Aviation Organization (ICAO) for air definition of “dangerous goods”. If the samples meet criteria for hazardous materials or dangerous goods, then DOT and IATA/ICAO regulations must be followed.

Soil samples preserved with methanol, and any excess methanol vials, must be shipped as "Dangerous Goods in Excepted Quantities" per the IATA regulations. The volume for excepted quantities of methanol is 30 mL per container and 300 mL per cooler. The class number is 3, flammable liquid, UN 1230.

Water samples preserved with hydrochloric acid or other de minimis amounts of preservative are not shipped as dangerous goods. However, excess pre-preserved sample containers with preservative must be shipped as "Dangerous Goods in Excepted Quantities" per the IATA regulations. The volume for excepted quantities of hydrochloric acid or nitric acid is 30 mL per container and 300 mL per cooler. The class number is 8, corrosive.

Samples that are being shipped as "Dangerous Goods in Excepted Quantities" must have the appropriate labeling and be declared as dangerous goods to the shipping carrier. However, no dangerous goods "candy-striped" form must be filled out and no Notification to Caption (NOTOC) is required.

Prior to shipping samples, complete the appropriate air waybill or manifest. Make sure to include the following:

- Laboratory name, address, and phone number
- Ahtna contact name, address, and phone number
- Project number
- Special handling requests

Keep a copy of the air waybill or manifest and submit it, with a copy of the COC, to the field lead or PM.

Upon shipping samples, notify the laboratory contact that samples are en route and provide an estimated arrival time.

6.0 REFERENCES

Alaska Department of Environmental Conservation (ADEC), 2017. *Field Sampling Guidance*, August.

International Air Transport Association (IATA), 2013. *Dangerous Goods Regulations*.

Code of Federal Regulations (CFR), 2013. Chapter 49, Parts 100-185.

7.0 REVISION LOG

Revision Date	Author	Revision Details
1/2/14	Brandie Hofmeister	Initial Issue
1/12/2015	Sara Perman	Removed wet ice shipping details.
6/2/16	Andrew Weller	2016 ADEC Field Sampling Guidance Update and Minor Grammar Edits
12/1/2016	Ashley Olson	Updated Logo

9/5/2017	Lexie Lucassen	Updated to Aug 2017 ADEC *Field Sampling Guidance*

(This Page Intentionally Left Blank)

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE FOR EQUIPMENT DECONTAMINATION No. 13

1.0 INTRODUCTION

1.1 Purpose

The purpose of this standard operating procedure (SOP) is to provide the step-by-step procedures for field decontamination of environmental sampling equipment and personal protective equipment (PPE) and applies to work conducted in modified Level D PPE.

Decontamination of equipment and PPE is designed to ensure that sample cross-contamination, human-health exposure, and contamination transport are minimized.

1.2 Scope

The scope of this SOP is to cover simple decontamination conducted by Ahtna personnel.

Simple decontamination procedures are generally applicable to field activities involving modified Level D PPE (steel toed boots, hard hat, safety glasses, and disposable nitrile gloves) where contact with hazardous substances is limited. Hazardous substances generally include petroleum products or other relatively benign chemical compounds. PPE decontamination is relatively straight forward under these circumstances.

The techniques described in this SOP are in general accordance with the Alaska Department of Environmental Conservation (ADEC) *Field Sampling Guidance*, dated August 2017.

2.0 RESPONSIBILITIES

Project Manager (PM) – The PM is responsible for providing adequate resources to the field staff and ensuring that field staff has adequate experience and training to successfully comply with the SOP. The PM is responsible for approving and documenting techniques that are not specifically described in this SOP but are considered the best sampling methods for the current project.

Site Safety and Health Officer (SSHO) – The SSHO oversees site-specific health and safety activities and ensures compliance with the project requirements. The SSHO conducts personal protective equipment (PPE) evaluations, selects the appropriate PPE, lists the requirements in the site-specific safety and health plan (SSHP), and coordinates with the field team to implement the

SSHP. SSHP provides guidance, ensures appropriate decontamination processes are implemented, and initiates corrective action.

Field Personnel – Field personnel are responsible for implementing the decontamination procedures outlined in this SOP and reporting and deficiencies.

3.0 DEFINITIONS

Decontamination Area – A location that is not expected to be contaminated and is upwind of suspected contaminants.

Exclusion Zone – A location designated to be used for decontamination of equipment and known to contain contaminated material.

Investigation-derived waste (IDW) – Waste that is generated in the process of investigation or examining a contaminated site.

Personal Protective Equipment (PPE) – Personal health and safety equipment used to protect the individual from contaminant exposure and physical injury.

4.0 EQUIPMENT

The following equipment is typically used for decontamination, but does not include all types of equipment that may be used.

- Sampling gloves
- Brushes, typically stiff bristle
- Buckets
- Laboratory-grade anionic detergent (Alconox, Liquinox, or similar)
- Spray or rinse bottles, or pump sprayer
- Paper towels
- Clean tap water
- Distilled water
- Garbage bags
- Plastic sheeting
- Waste containers

5.0 PROCEDURE

All non-disposable sampling equipment used at the site should be decontaminated both before activities begin and after each sample is collected. Drilling and excavation equipment shall be decontaminated prior to beginning site activities, at the termination of site activities, and, if used for sampling, prior to each sampling event.

5.1 Decontamination Area

Identify a localized decontamination area for drill rigs and other sampling equipment. Select the decontamination area so that decontamination fluids and soil wastes can be managed in a controlled area with minimal risk to the surrounding environment. The decontamination area should be large enough to allow temporary storage of cleaned equipment and materials before use, as well as to stage drums of decontamination investigation-derived waste (IDW). In the case of large decontamination areas (for example, for hollow-stem auger decontamination), line each area with a heavy-gauge plastic sheeting and include a collection system designed to capture potential decontamination IDW.

Decontamination areas should be laid out in such a way as to prevent overspray while performing equipment and personnel decontamination.

Smaller decontamination tasks, such as surface water and sediment equipment decontamination, may take place at the sampling locations. In this case, all required decontamination supplies and equipment must be mobilized to the site and smaller decontamination areas for personnel and portable equipment will be provided as necessary. These locations will include basins or tubs to capture decontamination IDW, which will be transferred to larger containers as necessary.

5.2 Personnel and Personal Protective Equipment

Personnel decontamination involves removal of gross contamination first. Contaminated solids such as mud should be scraped and wiped from boots, and gloves should be removed by rolling off the hands starting at the cuff in such a way that the gloves are turned inside out during removal. If necessary, a clean pair of gloves should be worn to complete the boot cleaning process. Boots can be cleaned while being worn or following removal. Any remaining contamination should be removed using soapy water, brushes or other similar means such as a pressure washer, if available. Once all debris is removed they should be rinsed with clean water. If boots are not laden with gross solid materials, a brush can simply be used to knock off or remove any residual solid materials. If the boots have contacted liquid phase contaminants, it is important that the contaminants be removed using soapy water and a brush followed by a clean water rinse. If the contaminants have adsorbed into the boots, the boots must be disposed of and a replacement pair obtained before conducting any further field activities.

Following removal and cleaning of reusable PPE, field personnel should wash their hands or any exposed body parts which may have been in contact with the associated hazardous substances.

5.3 Sampling Equipment Decontamination

All non-disposable sampling equipment should be cleaned prior to use. The following step by step procedure should be followed:

1. Remove as much gross contamination (such as pieces of soil) as possible off equipment at the sampling site.

2. If heavy petroleum residuals are encountered during sampling, an appropriate solvent such as methanol should be used to remove any petroleum residues from sampling equipment.
3. Wash water-resistant equipment thoroughly and vigorously with potable water containing laboratory-grade detergent such as Liquinox, Alconox, or equivalent, and using a bristle brush or similar utensil to remove any remaining residual contamination.
4. Rinse equipment thoroughly with potable water (1st rinse).
5. Rinse equipment thoroughly with distilled or deionized water (2nd rinse).
6. For sensitive field instruments, rinse equipment with distilled, deionized, or reagent grade water (3rd rinse).
7. Air dry at a location where dust or other fugitive contaminants may not contact the sample equipment. Alternatively, wet equipment maybe dried with a clean, disposable paper towel to assist the drying process. All equipment should be dry before reuse.

Clean, dry sampling equipment should be stored within a protective medium (plastic bag, etc.) or staged in a clean area for future use.

Cleaning and decontamination of the equipment should be accomplished in stages and in such a way that the contamination does not discharge into the environment. With CSP approval, decontamination water may be filtered on-site and reapplied directly to the ground surface within site boundaries at a minimum of 100 feet away from any drinking water wells and/or surface water bodies. Cleaning and decontamination wastes must be properly contained and disposed of in accordance with applicable local, state and federal regulations, as well as the CSP-approved work plan.

Disposable sampling equipment should be used whenever possible (e.g. drum thieves, bailers, spoons, etc.) to minimize the need to decontaminate these items.

5.4 Heavy Equipment Decontamination

Gross decontamination of equipment shall be performed whenever transporting or walking equipment within different areas of or between contaminated areas or exclusion zones. Gross decontamination shall focus on minimizing the spread of contaminated media as a result of equipment movement or transport. This decontamination process shall use dry methods (brooms, wipes, shovels, etc.) within the exclusion zone in order to remove large, easily dislodged deposits of soil and other contaminated media prior to exiting the exclusion zone. The site manager may increase the level of gross decontamination based upon the effectiveness of the use of dry decontamination.

When equipment is no longer needed on site and will be removed permanently from the site, it shall be decontaminated using brushes and/or a pressure washer with a detergent wash followed by a fresh water rinse. All areas of the equipment which were potentially contaminated shall be decontaminated as described in Section 5.3. Final decontamination shall occur within a decontamination pad in order to allow for the collection of decontamination materials, sludge, and water.

5.5 Dry Decontamination

In cases where dry decontamination is required, the following steps shall be followed at the sampling site:

1. Remove as much debris or contamination as possible using a dry brush or paper towel.
2. Spray equipment with a detergent/water mix.
3. Wipe down with a clean, dry paper towel.
4. Spray equipment with potable water.
5. Wipe down with a clean, dry paper towel.
6. Spray equipment with deionized or distilled water.
7. Wipe down with a clean, dry paper towel.

Dispose of all paper towels with other IDW and disposable sampling supplies.

6.0 REFERENCES

Alaska Department of Environmental Conservation (ADEC), 2017. *Field Sampling Guidance*, August.

ASTM, 2008. *Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*, Standard D5088-02.

7.0 REVISION LOG

Revision Date	Author	Revision Details
1/2/14	Brandie Hofmeister	Initial Issue
6/7/16	Andrew Weller	2016 ADEC Field Sampling Guidance Update
2/23/17	Katelyn Barnett Decker	Logo
9/5/2017	Lexie Lucassen	Updated ADEC *Field Sampling Guidance* to August 2017

(This Page Intentionally Left Blank)

Ahtna
Engineering Services, LLC

STANDARD OPERATING PROCEDURE
FOR PORE WATER SAMPLING
No. 34

1.0 PURPOSE/SCOPE

The purpose of this Standard Operating Procedure (SOP) is to direct field personnel in the techniques and requirements for collecting pore water samples.

The methodologies discussed in this SOP are applicable to the sampling of pore water in both flowing and standing water environments. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by site conditions and equipment limitations. However, if modifications occur, they should be documented in a site or personal logbook and discussed in reports summarizing field activities and analytical results.

2.0 RESPONSIBILITIES

Project Manager (**PM**) – The PM is responsible for providing adequate resources to the field staff and ensuring that field staff has adequate experience and training to successfully comply with the SOP. The PM is responsible for approving and documenting techniques that are not specifically described in this SOP, but are considered the best sampling methods for the current project.

Site Safety and Health Officer (SSHO) – The SSHO oversees site-specific health and safety activities and ensures compliance with the project requirements. The SSHO conducts personal protective equipment (PPE) evaluations, selects the appropriate PPE, lists the requirements in the site-specific safety and health plan (SSHP), and coordinates with the field team to implement the SSHP.

Field Team Lead (FTL)/Sampler – The FTL/sampler is responsible for the collection of samples in accordance with this in this SOP and the project specific work plan.

3.0 METHOD SUMMARY

Pore water samples may be collected using a variety of methods and equipment, depending on the physical location, contaminants present, and sediment type.

Pore water is collected through the use of a pore water extracting device; the most common of which is a stainless steel PushPoint™ sampler. The sampler typically consists of a cylindrical stainless steel tube with a screened zone and point at one end, and a sampling port at the other. Once the guard rod is positioned inside the sampler, the sampling end is inserted into the sediment

to the desired depth (as described in the project-specific work plan) with the use of a pressing/twisting motion. The sampling technician should take care to not create space around the sampling end that would allow for the intrusion of surface water as it could affect the sample results. Once in place, the guard rod can be removed and pore water extracted through the use of a syringe or peristaltic pump.

4.0 EQUIPMENT

- Pore water extracting device (PushPoint™ or similar). Length of screened zone and additional filters will be dependent upon individual project requirements.
- Syringe or peristaltic pump
- Teflon® tubing
- Laboratory-supplied sample containers, preservatives, labels, chain-of custody, custody seals, and temperature blanks.
- Collection containers for purged water.
- Bound field logbook with consecutive page numbers and waterproof, indelible pens/markers.
- Sampling gloves.
- Ice (gel ice or wet ice).
- PPE.

5.0 PROCEDURE

A new pair of plastic gloves are to be worn at each sampling location. All sampling equipment must be decontaminated prior to use. The location of each sample must be recorded prior to collecting the sample.

5.1 Pre-sampling Tasks

1. Insert guide rod into sampling tube
2. Press sampling tube into sediment below surface water to desired depth (ensure no gaps are created adjacent to the sampler that would allow the intrusion of surface water to sampling screen).
3. Remove guide rod (do not insert guide rod again until sampler and guide rod have been thoroughly cleaned).
4. Attach Teflon® tubing to sampling port of pore water extracting device.
5. Attach tubing to sample withdrawing device (syringe or peristaltic pump).

5.2 Sampling with a Syringe

1. Connect Teflon® tubing to syringe with three-way valve and a side syringe.
2. Switch valve to side syringe and purge air and ambient surface water (volume required for purge will be dependent upon size of the sampler and tubing).
3. Switch valve to sampling syringe and draw sample into syringe.
4. Syringe can be used as sample container or transfer sample to appropriate containers depending on project specific requirements.

5.3 Sampling with a Peristaltic Pump

When sampling with a peristaltic pump, it is possible for the pump to agitate the sample; it is because of this that there are two possible approaches for sampling with a peristaltic pump.

5.3.1 Standard Sampling with Peristaltic Pump

1. Connect Teflon® tubing to the pump head tubing.
2. Purge the pore water sampling device at a low flow (50-200 mL per minute). Water is assumed to be initially turbid.
3. When the water has cleared, continue to purge a volume determined by project specific requirements.
4. Collect samples directly from pump tubing into appropriate containers depending on project specific requirements.

5.3.2 Low Agitation Sampling with Peristaltic Pump

1. Connect Teflon® tubing to the pump head tubing.
2. Purge the pore water sampling device at a low flow (50-200 mL per minute). Water is assumed to be initially turbid.
3. When the water has cleared, continue to purge a volume determined by project specific requirements.
4. Once tubing is full of water, suspend pumping and disconnect tubing from pore water sampler.
5. Reverse the peristaltic pump at very low speed and collect samples into appropriate containers depending on project specific requirements.

5.4 Sample Preservation, Containers, Handling and Storage

- During sample collection, special care should be taken to ensure the sampling device does not come in contact with the sample containers.
- Samples should be clearly labeled and placed into appropriate containers per project-specific requirements.
- Samples collected for volatile organic compound (VOC) analysis must not have any headspace.
- All samples requiring preservation should be preserved as soon as practically possible after collection and be stored with trip blanks, as required.

6.0 REFERENCES

- US EPA. 1984. *Characterization of hazardous waste- a methods manual: Volume II: Available sampling methods, second edition. EPA-600/4-84-076.*
- Environmental Protection Agency (EPA), 2007. *Pore Water Sampling, SESD Operating Procedure, SESDPROC-513-R3*. Last revised 16 December, 2016.
- Zimmerman, M.J., Massey, A.M., and Campo, K.W., 2005, *Pushpoint sampling for defining spatial and temporal variations in contaminant concentrations in sediment pore*

water near the ground-water/surface-water interface: U.S. Geological Survey Scientific Investigations Report 2005-5036, 70 p.

7.0 REVISION LOG

Revision Date	Author	Revision Details
5/30/18	Baley Lenhart	Initial Issue

Appendix B – Laboratory Quality Control Information

(This Page Intentionally Left Blank)

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Roxanne Sullivan Phone: 303-736-0100
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2019 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.1 of the DoD Quality Systems Manual for Environmental Laboratories), 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

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Antimony	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Barium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Boron	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Calcium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Chromium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Copper	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Iron	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Lead	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Lithium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Magnesium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Manganese	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Mercury	EPA 200.7	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Nickel	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Potassium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Selenium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Silica	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Silicon	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Silver	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Sodium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Strontium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Thallium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Tin	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Titanium	EPA 200.7	EPA 6010B/6010C	EPA 6010B/6010C
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Zinc	EPA 200.7/200.8	EPA 6010B/6010C EPA 6020/6020A	EPA 6010B/6010C EPA 6020/6020A
Nutrients			
Nitrate (as N)	By calculation	EPA 300.0 EPA 9056/9056A By calculation	EPA 9056/9056A By calculation
Nitrate-nitrite (as N)	EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A
Orthophosphate (as P)	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	----------	EPA 6010B/6010C	EPA 6010B/6010C
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinty)	SM 2320B-1997	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Hexavalent chromium	----------	EPA 7196A	----------
pH	----------	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	EPA 9071B
Percent moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, total	SM 2540B	SM 2540B	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Sulfide, total	----------	EPA 9034	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	----------	EPA 351.2	----------
Purgeable Organics (volatiles)			
Acetone	EPA 624	EPA 8260B	EPA 8260B
Acetonitrile	----------	EPA 8260B	EPA 8260B
Acrolein	EPA 624	EPA 8260B	EPA 8260B
Acrylonitrile	EPA 624	EPA 8260B	EPA 8260B
Allyl Chloride	----------	EPA 8260B	EPA 8260B
tert-Amyl Methyl Ether			
Benzene	EPA 624	EPA 8260B/8260B SIM EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/OK DEQ GRO
Bromobenzene	----------	EPA 8260B	EPA 8260B
Bromochloromethane	----------	EPA 8260B	EPA 8260B
Bromodichloromethane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Bromoform	EPA 624	EPA 8260B	EPA 8260B
Bromomethane	EPA 624	EPA 8260B	EPA 8260B
Butadiene	----------	EPA 8260B SIM	EPA 8260B SIM
2-Butanone	EPA 624	EPA 8260B	EPA 8260B
n-Butyl alcohol	----------	EPA 8260B EPA 8015B/8015C	EPA 8260B EPA 8015B/8015C
tert-Butyl alcohol (2-Methyl-2-propanol)	----------	EPA 8260B EPA 8260B SIM	EPA 8260B
n-Butylbenzene	----------	EPA 8260B	EPA 8260B
sec-Butylbenzene	----------	EPA 8260B	EPA 8260B
tert-Butylbenzene	----------	EPA 8260B	EPA 8260B
Carbon disulfide	EPA 624	EPA 8260B	EPA 8260B
Carbon tetrachloride	EPA 624	EPA 8260B	EPA 8260B
Chlorobenzene	EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA 8021B
2-Chloro-1,3-butadiene	----------	EPA 8260B	EPA 8260B
Chloroethane	EPA 624	EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	EPA 624	EPA 8260B	EPA 8260B
Chloroform	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
1-Chlorohexane	----------	EPA 8260B	EPA 8260B
Chloromethane	EPA 624	EPA 8260B	EPA 8260B
Chloroprene	----------	EPA 8260B	EPA 8260B
4-Chlorotoluene	----------	EPA 8260B	EPA 8260B
2-Chlorotoluene	----------	EPA 8260B	EPA 8260B
Cyclohexane	----------	EPA 8260B	EPA 8260B
Cyclohexanone	----------	EPA 8260B	EPA 8260B
Dibromochloromethane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624	EPA 8260B EPA 8011	EPA 8260B EPA 8011
Dibromochloromethane	----------	EPA 8260B	EPA 8260B
Dichlorodifluoromethane	EPA 624	EPA 8260B	EPA 8260B
Dibromomethane	EPA 624	EPA 8260B	EPA 8260B
1,2 Dibromoethane (EDB)	EPA 624	EPA 8260B EPA 8011	EPA 8260B EPA 8011
1,2-Dichlorobenzene	EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA 8021B
1,3-Dichlorobenzene	EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA 8021B
1,4-Dichlorobenzene	EPA 624	EPA 8260B EPA 8021B	EPA 8260B EPA8021B
cis-1,4-Dichloro-2-butene	----------	EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene	----------	EPA 8260B	EPA 8260B
1,1-Dichloroethane	EPA 624	EPA 8260B	EPA 8260B
1,2-Dichloroethane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
1,1-Dichloroethene	EPA 624	EPA 8260B	EPA 8260B
1,2-Dichloroethene	EPA 624	EPA 8260B	EPA 8260B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
cis-1,2-Dichloroethene	EPA 624	EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene	EPA 624	EPA 8260B	EPA 8260B
Dichlorofluoromethane	----------	EPA 8260B	EPA 8260B
1,2-Dichloropropane	EPA 624	EPA 8260B	EPA 8260B
1,3-Dichloropropane	----------	EPA 8260B	EPA 8260B
2,2-Dichloropropane	----------	EPA 8260B	EPA 8260B
1,1-Dichloropropene	----------	EPA 8260B	EPA 8260B
1,3-Dichloropropene	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
cis-1,3-Dichloropropene	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
trans-1,3-Dichloropropene	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Diethyl ether	----------	EPA 8260B	EPA 8260B
Di-isopropylether	----------	EPA 8260B	EPA 8260B
1,4-Dioxane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Ethanol	----------	EPA 8260B EPA 8015B/8015C	EPA 8260B EPA 8015B/8015C
Ethyl acetate	----------	EPA 8260B	EPA 8260B
Ethyl benzene	EPA 624	EPA 8260B EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/OK DEQ GRO
Ethyl methacrylate	----------	EPA 8260B	EPA 8260B
Ethyl tert-butyl ether	----------	----------	----------
Ethylene glycol	----------	EPA 8015C	EPA 8015C
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO
Hexane	EPA 624	EPA 8260B	EPA 8260B
2-Hexanone	EPA 624	EPA 8260B	EPA 8260B
Hexachlorobutadiene	----------	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B EPA 8015B/8015C	EPA 8260B EPA 8015B/8015C
Isopropyl alcohol	----------	EPA 8260B	EPA 8260B
Isopropylbenzene	----------	EPA 8260B	EPA 8260B
1,4-Isopropyltoluene	----------	EPA 8260B	EPA 8260B
Iodomethane	----------	EPA 8260B	EPA 8260B
Methacrylonitrile	----------	EPA 8260B	EPA 8260B
Methanol	----------	EPA 8015B/8015C	EPA 8015B/8015C
Methyl acetate	----------	EPA 8260B	EPA 8260B
Methyl cyclohexane	----------	EPA 8260B	EPA 8260B
Methylene chloride	EPA 624	EPA 8260B	EPA 8260B
Methyl ethyl ketone (MEK)	----------	EPA 8260B	EPA 8260B
Methyl isobutyl ketone	----------	EPA 8260B	EPA 8260B
Methyl methacrylate	----------	EPA 8260B	EPA 8260B
Methyl tert-butyl ether (MtBE)	EPA 624	EPA 8260B EPA 8021B OK DEQ GRO	EPA 8260B EPA 8021B OK DEQ GRO

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
4-Methyl-2-pentanone (MIBK)	EPA 624	EPA 8260B	EPA 8260B
Naphthalene	EPA 624	EPA 8260B OK DEQ GRO	EPA 8260B OK DEQ GRO
2-Nitropropane	----------	EPA 8260B	EPA 8260B
2,2' Oxybisethanol	----------	EPA 8015C	EPA 8015C
2-Pentanone	----------	EPA 8260B	EPA 8260B
Propionitrile	----------	EPA 8260B	EPA 8260B
n-Propylbenzene	----------	EPA 8260B	EPA 8260B
Propylene glycol	----------	EPA 8015C	EPA 8015C
Styrene	EPA 624	EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	EPA 624	EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Tetrachloroethene	EPA 624	EPA 8260B	EPA 8260B
Tetrahydrofuran	----------	EPA 8260B	EPA 8260B
Toluene	EPA 624	EPA 8260B EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
1,2,3-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	EPA 624	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Trichloroethene	EPA 624	EPA 8260B/8260B SIM	EPA 8260B
Trichlorofluoromethane	EPA 624	EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	EPA 624	EPA 8260B EPA 8011	EPA 8260B EPA 8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B	EPA 8260B
Triethylene glycol	----------	EPA 8015C	EPA 8015C
1,2,3-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
1,2,4-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
Vinyl acetate	EPA 624	EPA 8260B	EPA 8260B
Vinyl chloride	EPA 624	EPA 8260B EPA 8260B SIM	EPA 8260B EPA 8260B SIM
Xylenes, total	EPA 624	EPA 8260B EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/OK DEQ GRO
1,2-Xylene (o-Xylene)	EPA 624	EPA 8260B EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/OK DEQ GRO
m+p-Xylene	EPA 624	EPA 8260B EPA 8021B AK101/OK DEQ GRO	EPA 8260B EPA 8021B AK101/ K DEQ GRO
Methane	----------	RSK-175	----------

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Ethane	----------	RSK-175	----------
Ethylene (Ethene)	----------	RSK-175	----------
Acetylene	----------	RSK-175	----------
Acetylene ethane	----------	RSK-175	----------
Extractable Organics (semivolatiles)			
Acenaphthene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acenaphthylene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acetophenone	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alachlor	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Anthracene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Aramite	----------	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde	----------	EPA 8270C 8270D	EPA 8270C/8270D
Benzidine	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Benzo(a)anthracene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(b)fluoranthene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(k)fluoranthene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(ghi)perylene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(a)pyrene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzyl alcohol	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethoxy) methane	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethyl) ether	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
bis (2-Chloroisopropyl) ether (2,2'Oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Ethylhexyl) phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
butyl Benzyl phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene	----------	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Chlorobenzilate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-chloro-3-Methylphenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
1-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Cresols	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diallate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,h) anthracene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Dibenzofuran	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
2,4-Dichlorophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D	EPA 8270C/8270D
alpha-,alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
di-n-butyl Phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
di-n-octyl Phthalate	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	----------	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
1,4-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
2,4-Dinitrotoluene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
2,6-Dinitrotoluene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
1,4-Dioxane	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/ AK102/8015D/OK DEQ DRO	EPA 8015B/8015C/ AK102/8015D/OK DEQ DRO
ethyl Methanesulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	----------	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	EPA 625	EPA 8270C/8270D EPA8270D SIM	EPA 8270C/8270D EPA 8270D SIM

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Fluorene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Hexachlorobenzene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Hexachlorobutadiene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Hexachloroethane	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Hexachloropropene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Isodrin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	----------	EPA 8270C/8270D	EPA 8270 C/8270D
Methapyrilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-methyl-4,6-Dinitrophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
methyl Methane sulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylphenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
1,4-Naphthoquinone	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
2-Nitrophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
N-Nitrosodiphenylamine	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
5-nitro-o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-chloropropane)	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, methyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	EPA 625	EPA 8270C/8270D EPA 8270C SIM/8270D SIM EPA 8321A/8321B	EPA 8270C/8270D EPA 8270C SIM/8270D SIM EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Phenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Phorate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	EPA 625	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Pyridine	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
Safrole	----------	EPA 8270C/8270D	EPA 8270C/8270D
Sulfotepp	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin	----------	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	EPA 625	EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Pesticides/Herbicides/PCBs			
Aldrin	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
beta-BHC	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
gamma-BHC	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-Chlordane	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4-D	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dalapon	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
2,4-DB	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
4,4'-DDD	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dieldrin	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb	----------	EPA 8151A EPA 8321A	EPA 8321A
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608	EPA 8081A /8081B	EPA 8081A/8081B
Endonsulfan sulfate	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
MCPP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA8321A
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608	EPA 8082/8082A	EPA 8082/8082A
Total PCBs	EPA 608	EPA 8082/8082A	EPA 8082/8082A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Toxaphene	EPA 608	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Diamino-4-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Diamino-6-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			

Parameter/Analyte	**Non-Potable Water**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	----------	EPA 9050A	EPA 9050A
Corrosivity	----------	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
Organic Prep Methods			
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Soxhlet extraction	----------	----------	EPA 3540C
Microwave extraction	----------	----------	EPA 3546
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Waste dilution	----------	EPA 3580A	EPA 3580A
Solid phase extraction	----------	EPA 3535A	----------
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Organic Cleanup Procedures			
Florisil cleanup	----------	EPA 3620B	EPA 3620B
Florisil cleanup	----------	EPA 3620C	EPA 3620C
Sulfur cleanup	----------	EPA 3660A	EPA 3660A
Sulfuric acid/Permanganate cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid digestion total recoverable or dissolved metals	----------	EPA 3005A	----------
Acid digestion for total metals	----------	EPA 3010A	----------
Acid digestion for total metals	----------	EPA 3020A	----------
Acid digestion of sediments, sludges and soils	----------	----------	EPA 3050B

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, 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:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	**Method**
Metals	
Cadmium	EPA 6010C
Chromium	EPA 6010C
Lead	EPA 6010C
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (volatiles)	
tert-Amyl Methyl Ether	EPA 8260B
Benzene	EPA 8260B EPA 8021B
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B
1,2-Dichloroethane	EPA 8260B
Di-isopropylether	EPA 8260B
Ethyl benzene	EPA 8260B EPA 8021B
Ethyl tert-butyl ether	EPA 8260B
Gas Range Organics (GRO)	EPA 8015C
Methyl tert-butyl ether (MTBE)	EPA 8260B EPA 8021B
Naphthalene	EPA 8260B EPA 8021B
Toluene	EPA 8260B EPA 8021B
Xylenes, total	EPA 8260B EPA 8021B
1,2-Xylene	EPA 8260B EPA 8021B
M+P-Xylene	EPA 8260B EPA 8021B
Extractable Organics (semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015C (WY: C10-C32)
Organic Prep Methods	
Volatiles purge and trap	EPA 5030B (water) /5030A (solids)

Accredited Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.1 of the DoD Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Presented this 30th day of October 2017.

President & CEO
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2019

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

THE STATE of ALASKA
GOVERNOR BILL WALKER

Department of Environmental Conservation

DIVISION OF SPILL PREVENTION AND RESPONSE
Contaminated Sites Program
Laboratory Approval Program

555 Cordova Street
Anchorage, Alaska 99501
Main: 907.465.5390
Fax: 907.269.7649
cs.lab.cert@alaska.gov

February 12, 2018

Dennis Bean
TestAmerica – Seattle
5755 8th Street East
Tacoma, WA 98424

RE: Contaminated Sites Laboratory Approval **17-024**

Dear Mr. Bean,

Thank you for submitting an application to the Alaska Department of Environmental Conservation's Contaminated Sites Laboratory Approval Program (CS-LAP), on October 31, 2017. Based on your lab's National Environmental Laboratory Accreditation Program (NELAP) approval through the Oregon Environmental Laboratory Accreditation Program (ORELAP), and Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) approval through the ANSI-ASQ National Accreditation Board (ANAB), TestAmerica – Seattle, located at the above address, is granted ***Approved*** status to perform the analyses listed in the attached *Scope of Approval*, for Alaska contaminated sites projects, including underground storage tanks and leaking underground storage tank sites (UST/LUST), under the July 1, 2017 amendments to 18 AAC 78. This approval expires on ***January 19, 2019***.

Be aware that **any** changes in your NELAP or DoD-ELAP approval status must be reported to the CS program within 3 business days. Failure to do so will result in revocation of **all** CS-LAP approvals for a period of one year. Notification should be in writing sent to cs.lab.cert@alaska.gov. We recommend also contacting the CS-LAP by telephone to verify that the message was received.

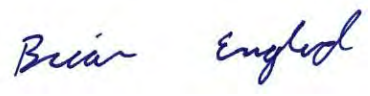
To report any changes in your lab's contact information (i.e. lab director, business name, location, etc.), please complete the form found at http://dec.alaska.gov/spar/csp/LabApproval/ApplyForApproval.htm and submit to cs.submittals@alaska.gov.

To apply for renewal of your approval, please complete the application found at http://dec.alaska.gov/spar/csp/LabApproval/ApplyForApproval.htm and submit to cs.submittals@alaska.gov. The required documentation must be submitted for renewal no later than 30 days before your date of expiration.

Please remember to include the laboratory's ID number, listed above, on all correspondence concerning the laboratory.

If you have any questions, please contact the CS-LAP at (907) 465-5390, or by email at cs.lab.cert@alaska.gov. Labs are also highly encouraged to join the CS-LAP listserv by going to http://list.state.ak.us/mailman/listinfo/cs.lab.approval.

Respectfully,



Brian Englund
Alaska CS Lab Approval Officer

Attachment: Scope of Approval

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Acenaphthene	83-32-9	625	---	**X**	---	ANAB
Acenaphthene	83-32-9	8270C	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270C-SIM	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270D	**X**	**X**	---	ANAB
Acenaphthene	83-32-9	8270D-SIM	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	625	---	**X**	---	ANAB
Acenaphthylene	208-96-8	8270C	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270C-SIM	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270D	**X**	**X**	---	ANAB
Acenaphthylene	208-96-8	8270D-SIM	**X**	**X**	---	ANAB
Acetone	67-64-1	624	---	**X**	---	ANAB
Acetone	67-64-1	8260B	**X**	**X**	---	ANAB
Acetone	67-64-1	8260C	**X**	**X**	---	ANAB
Aldrin	309-00-2	608	---	**X**	---	ANAB
Aldrin	309-00-2	8081A	**X**	**X**	---	ANAB
Aldrin	309-00-2	8081B	**X**	**X**	---	ANAB
Anthracene	120-12-7	625	---	**X**	---	ANAB
Anthracene	120-12-7	8270C	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270C-SIM	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270D	**X**	**X**	---	ANAB
Anthracene	120-12-7	8270D-SIM	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	200.7	---	**X**	---	ANAB
Antimony (metallic)	7440-36-0	200.8	---	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6010B	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6010C	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6020A	**X**	**X**	---	ANAB
Antimony (metallic)	7440-36-0	6020B	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Arsenic, Inorganic	7440-38-2	200.7	---	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	200.8	---	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6010B	**X**	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6010C	**X**	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6020A	**X**	**X**	---	ANAB
Arsenic, Inorganic	7440-38-2	6020B	**X**	**X**	---	ANAB
Barium	7440-39-3	200.7	---	**X**	---	ANAB
Barium	7440-39-3	200.8	---	**X**	---	ANAB
Barium	7440-39-3	6010B	**X**	**X**	---	ANAB
Barium	7440-39-3	6010C	**X**	**X**	---	ANAB
Barium	7440-39-3	6020A	**X**	**X**	---	ANAB
Barium	7440-39-3	6020B	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	625	---	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270C	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270C-SIM	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270D	**X**	**X**	---	ANAB
Benz[a]anthracene	56-55-3	8270D-SIM	**X**	**X**	---	ANAB
Benzene	71-43-2	624	---	**X**	---	ANAB
Benzene	71-43-2	8260B	**X**	**X**	---	ANAB
Benzene	71-43-2	8260B-SIM	**X**	**X**	---	ANAB
Benzene	71-43-2	8260C	**X**	**X**	---	ANAB
Benzene	71-43-2	8260C-SIM	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	625	---	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270C	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270C-SIM	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270D	**X**	**X**	---	ANAB
Benzo[a]pyrene	50-32-8	8270D-SIM	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Benzo[b]fluoranthene	205-99-2	625	---	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270C	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270C-SIM	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270D	**X**	**X**	---	ANAB
Benzo[b]fluoranthene	205-99-2	8270D-SIM	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	625	---	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270C	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270C-SIM	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270D	**X**	**X**	---	ANAB
Benzo[g,h,i]perylene	191-24-2	8270D-SIM	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	625	---	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270C	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270C-SIM	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270D	**X**	**X**	---	ANAB
Benzo[k]fluoranthene	207-08-9	8270D-SIM	**X**	**X**	---	ANAB
Benzoic Acid	65-85-0	8270C	**X**	**X**	---	ANAB
Benzoic Acid	65-85-0	8270D	**X**	**X**	---	ANAB
Benzyl Alcohol	100-51-6	8270C	**X**	**X**	---	ANAB
Benzyl Alcohol	100-51-6	8270D	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	200.7	---	**X**	---	ANAB
Beryllium and compounds	7440-41-7	200.8	---	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6010B	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6010C	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6020A	**X**	**X**	---	ANAB
Beryllium and compounds	7440-41-7	6020B	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270C	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270C-SIM	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Bis(2-chloroethyl)ether	111-44-4	8270D	**X**	**X**	---	ANAB
Bis(2-chloroethyl)ether	111-44-4	8270D-SIM	**X**	**X**	---	ANAB
Bis(2-ethylhexyl)phthalate (DEHP)	117-81-7	8270C	**X**	**X**	---	ANAB
Bis(2-ethylhexyl)phthalate (DEHP)	117-81-7	8270D	**X**	**X**	---	ANAB
Bromobenzene	108-86-1	624	---	**X**	---	ANAB
Bromobenzene	108-86-1	8260B	**X**	**X**	---	ANAB
Bromobenzene	108-86-1	8260C	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	624	---	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260B	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260B-SIM	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260C	**X**	**X**	---	ANAB
Bromodichloromethane	75-27-4	8260C-SIM	**X**	**X**	---	ANAB
Bromoform	75-25-2	624	---	**X**	---	ANAB
Bromoform	75-25-2	8260B	**X**	**X**	---	ANAB
Bromoform	75-25-2	8260B-SIM	**X**	**X**	---	ANAB
Bromoform	75-25-2	8260C	**X**	**X**	---	ANAB
Bromoform	75-25-2	8260C-SIM	**X**	**X**	---	ANAB
Bromomethane	74-83-9	624	---	**X**	---	ANAB
Bromomethane	74-83-9	8260B	**X**	**X**	---	ANAB
Bromomethane	74-83-9	8260B-SIM	**X**	**X**	---	ANAB
Bromomethane	74-83-9	8260C	**X**	**X**	---	ANAB
Bromomethane	74-83-9	8260C-SIM	**X**	**X**	---	ANAB
Butadiene, 1,3-	106-99-0	8260B-SIM	**X**	**X**	---	ORELAP
Butadiene, 1,3-	106-99-0	8260C-SIM	**X**	**X**	---	ORELAP
Butanol, N-	71-36-3	8260B	**X**	**X**	---	ORELAP
Butanol, N-	71-36-3	8260C	**X**	**X**	---	ORELAP
Butyl Benzyl Phthalate	85-68-7	8270C	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Butyl Benzyl Phthalate	85-68-7	8270D	**X**	**X**	---	ANAB
Butylbenzene, n-	104-51-8	624	---	**X**	---	ANAB
Butylbenzene, n-	104-51-8	8260B	**X**	**X**	---	ANAB
Butylbenzene, n-	104-51-8	8260C	**X**	**X**	---	ANAB
Butylbenzene, sec-	135-98-8	624	---	**X**	---	ANAB
Butylbenzene, sec-	135-98-8	8260B	**X**	**X**	---	ANAB
Butylbenzene, sec-	135-98-8	8260C	**X**	**X**	---	ANAB
Butylbenzene, tert-	98-06-6	624	---	**X**	---	ANAB
Butylbenzene, tert-	98-06-6	8260B	**X**	**X**	---	ANAB
Butylbenzene, tert-	98-06-6	8260C	**X**	**X**	---	ANAB
Cadmium	7440-43-9	200.7	---	**X**	---	ANAB
Cadmium	7440-43-9	200.8	---	**X**	---	ANAB
Cadmium	7440-43-9	6010B	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6010C	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6020A	**X**	**X**	---	ANAB
Cadmium	7440-43-9	6020B	**X**	**X**	---	ANAB
Carbon Disulfide	75-15-0	624	---	**X**	---	ANAB
Carbon Disulfide	75-15-0	8260B	**X**	**X**	---	ANAB
Carbon Disulfide	75-15-0	8260C	**X**	**X**	---	ANAB
Carbon Tetrachloride	56-23-5	624	---	**X**	---	ANAB
Carbon Tetrachloride	56-23-5	8260B	**X**	**X**	---	ANAB
Carbon Tetrachloride	56-23-5	8260C	**X**	**X**	---	ANAB
Chlordane, Total	12789-03-6	608	---	**X**	---	ANAB
Chlordane, Total	12789-03-6	8081A	**X**	**X**	---	ANAB
Chlordane, Total	12789-03-6	8081B	**X**	**X**	---	ANAB
Chlordane, α-	5103-71-9	608	---	**X**	---	ANAB
Chlordane, α-	5103-71-9	8081A	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Chlordane, α-	5103-71-9	8081B	**X**	**X**	---	ANAB
Chlordane, γ-	5103-74-2	608	---	**X**	---	ANAB
Chlordane, γ-	5103-74-2	8081A	**X**	**X**	---	ANAB
Chlordane, γ-	5103-74-2	8081B	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270C	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270D	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270C-SIM	**X**	**X**	---	ANAB
Chloroaniline, p-	106-47-8	8270D-SIM	**X**	**X**	---	ANAB
Chlorobenzene	108-90-7	624	---	**X**	---	ANAB
Chlorobenzene	108-90-7	8260B	**X**	**X**	---	ANAB
Chlorobenzene	108-90-7	8260C	**X**	**X**	---	ANAB
Chloroform	67-66-3	624	---	**X**	---	ANAB
Chloroform	67-66-3	8260B	**X**	**X**	---	ANAB
Chloroform	67-66-3	8260B-SIM	**X**	**X**	---	ANAB
Chloroform	67-66-3	8260C	**X**	**X**	---	ANAB
Chloroform	67-66-3	8260C-SIM	**X**	**X**	---	ANAB
Chloromethane	74-87-3	624	---	**X**	---	ANAB
Chloromethane	74-87-3	8260B	**X**	**X**	---	ANAB
Chloromethane	74-87-3	8260C	**X**	**X**	---	ANAB
Chloronaphthalene, Beta-	91-58-7	625	---	**X**	---	ANAB
Chloronaphthalene, Beta-	91-58-7	8270C	**X**	**X**	---	ANAB
Chloronaphthalene, Beta-	91-58-7	8270D	**X**	**X**	---	ANAB
Chlorophenol, 2-	91-58-7	625	---	**X**	---	ANAB
Chlorophenol, 2-	95-57-8	8270C	**X**	**X**	---	ANAB
Chlorophenol, 2-	95-57-8	8270D	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	200.7	---	**X**	---	ANAB
Chromium (Total)	7440-47-3	200.8	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Chromium (Total)	7440-47-3	6010B	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	6010C	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	6020A	**X**	**X**	---	ANAB
Chromium (Total)	7440-47-3	6020B	**X**	**X**	---	ANAB
Chromium (VI)	18540-29-9	SM 3500-CR B	---	**X**	---	ANAB
Chrysene	218-01-9	625	---	**X**	---	ANAB
Chrysene	218-01-9	8270C	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270C-SIM	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270D	**X**	**X**	---	ANAB
Chrysene	218-01-9	8270D-SIM	**X**	**X**	---	ANAB
Copper	7440-50-8	200.7	---	**X**	---	ANAB
Copper	7440-50-8	200.8	---	**X**	---	ANAB
Copper	7440-50-8	6010B	**X**	**X**	---	ANAB
Copper	7440-50-8	6010C	**X**	**X**	---	ANAB
Copper	7440-50-8	6020A	**X**	**X**	---	ANAB
Copper	7440-50-8	6020B	**X**	**X**	---	ANAB
Cresol, o- (2-Methylphenol)	95-48-7	625	---	**X**	---	ANAB
Cresol, o- (2-Methylphenol)	95-48-7	8270C	**X**	**X**	---	ANAB
Cresol, o- (2-Methylphenol)	95-48-7	8270D	**X**	**X**	---	ANAB
Cumene (Isopropylbenzene)	98-82-8	8260B	**X**	**X**	---	ANAB
Cumene (Isopropylbenzene)	98-82-8	8260C	**X**	**X**	---	ANAB
DDD, 4,4'-	72-54-8	608	---	**X**	---	ANAB
DDD, 4,4'-	72-54-8	8081A	**X**	**X**	---	ANAB
DDD, 4,4'-	72-54-8	8081B	**X**	**X**	---	ANAB
DDE, 4,4'-	72-55-9	608	---	**X**	---	ANAB
DDE, 4,4'-	72-55-9	8081A	**X**	**X**	---	ANAB
DDE, 4,4'-	72-55-9	8081B	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
DDT, 4,4'-	50-29-3	608	---	**X**	---	ANAB
DDT, 4,4'-	50-29-3	8081A	**X**	**X**	---	ANAB
DDT, 4,4'-	50-29-3	8081B	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	625	---	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270C	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270C-SIM	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270D	**X**	**X**	---	ANAB
Dibenz[a,h]anthracene	53-70-3	8270D-SIM	**X**	**X**	---	ANAB
Dibenzofuran	132-64-9	8270C	**X**	**X**	---	ANAB
Dibenzofuran	132-64-9	8270D	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	624	---	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260B	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260B-SIM	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260C	**X**	**X**	---	ANAB
Dibromochloromethane	124-48-1	8260C-SIM	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	624	---	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8011	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260B	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260B-SIM	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260C	**X**	**X**	---	ANAB
Dibromoethane, 1,2- (Ethylene Dibromide)	106-93-4	8260C-SIM	**X**	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	624	---	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260B	**X**	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260B-SIM	**X**	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260C	**X**	**X**	---	ANAB
Dibromomethane (Methylene Bromide)	74-95-3	8260C-SIM	**X**	**X**	---	ANAB
Dibutyl Phthalate	84-74-2	625	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dibutyl Phthalate	84-74-2	8270C	**X**	**X**	---	ANAB
Dibutyl Phthalate	84-74-2	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	624	---	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	625	---	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8260B	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8260C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,2-	95-50-1	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	624	---	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	625	---	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	8260B	**X**	**X**	---	ORELAP
Dichlorobenzene, 1,3-	541-73-1	8260C	**X**	**X**	---	ORELAP
Dichlorobenzene, 1,3-	541-73-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,3-	541-73-1	8270D	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	624	---	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	625	---	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260B	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260B-SIM	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8260C-SIM	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8270C	**X**	**X**	---	ANAB
Dichlorobenzene, 1,4-	106-46-7	8270D	**X**	**X**	---	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270C	**X**	**X**	---	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270C-SIM	**X**	---	---	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270D	**X**	**X**	---	ANAB
Dichlorobenzidine, 3,3'-	91-94-1	8270D-SIM	**X**	---	---	ANAB
Dichlorodifluoromethane	75-71-8	624	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dichlorodifluoromethane	75-71-8	8260B	**X**	**X**	---	ANAB
Dichlorodifluoromethane	75-71-8	8260C	**X**	**X**	---	ANAB
Dichloroethane, 1,1-	75-34-3	624	---	**X**	---	ANAB
Dichloroethane, 1,1-	75-34-3	8260B	**X**	**X**	---	ANAB
Dichloroethane, 1,1-	75-34-3	8260C	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	624	---	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	8260B	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	8260B-SIM	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	8260C	**X**	**X**	---	ANAB
Dichloroethane, 1,2-	107-06-2	8260C-SIM	**X**	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	624	---	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	8260B-SIM		**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	8260C	**X**	**X**	---	ANAB
Dichloroethylene, 1,1-	75-35-4	8260C-SIM	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	624	---	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260B-SIM	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260C	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-cis-	156-59-2	8260C-SIM	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	624	---	**X**	---	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	8260B	**X**	**X**	---	ANAB
Dichloroethylene, 1,2-trans-	156-60-5	8260C	**X**	**X**	---	ANAB
Dichlorophenol, 2,4-	120-83-2	625	---	**X**	---	ANAB
Dichlorophenol, 2,4-	120-83-2	8270C	**X**	**X**	---	ANAB
Dichlorophenol, 2,4-	120-83-2	8270D	**X**	**X**	---	ANAB
Dichlorophenoxy Acetic Acid, 2,4-	94-75-7	8151A	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dichloropropane, 1,2-	78-87-5	624	---	**X**	---	ANAB
Dichloropropane, 1,2-	78-87-5	8260B	**X**	**X**	---	ANAB
Dichloropropane, 1,2-	78-87-5	8260C	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260B	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260B-SIM	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260C	**X**	**X**	---	ANAB
Dichloropropene, 1,3- (cis + trans)	542-75-6	8260C-SIM	**X**	**X**	---	ANAB
Dieldrin	60-57-1	608	---	**X**	---	ANAB
Dieldrin	60-57-1	8081A	**X**	**X**	---	ANAB
Dieldrin	60-57-1	8081B	**X**	**X**	---	ANAB
Diethyl Phthalate	84-66-2	625	---	**X**	---	ANAB
Diethyl Phthalate	84-66-2	8270C	**X**	**X**	---	ANAB
Diethyl Phthalate	84-66-2	8270D	**X**	**X**	---	ANAB
Dimethylphenol, 2,4-	105-67-9	625	---	**X**	---	ANAB
Dimethylphenol, 2,4-	105-67-9	8270C	**X**	**X**	---	ANAB
Dimethylphenol, 2,4-	105-67-9	8270D	**X**	**X**	---	ANAB
Dimethylphthalate	131-11-3	625	---	**X**	---	ANAB
Dimethylphthalate	131-11-3	8270C	**X**	**X**	---	ANAB
Dimethylphthalate	131-11-3	8270D	**X**	**X**	---	ANAB
Dinitrobenzene, 1,3-	99-65-0	8270C-SIM	**X**	**X**	---	ANAB
Dinitrobenzene, 1,3-	99-65-0	8270D-SIM	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	625	---	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270C	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270C-SIM	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270D	**X**	**X**	---	ANAB
Dinitrophenol, 2,4-	51-28-5	8270D-SIM	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	625	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Dinitrotoluene, 2,4-	121-14-2	8270C	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8270C-SIM	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8270D	**X**	**X**	---	ANAB
Dinitrotoluene, 2,4-	121-14-2	8270D-SIM	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	625	---	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270C	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270C-SIM	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270D	**X**	**X**	---	ANAB
Dinitrotoluene, 2,6-	606-20-2	8270D-SIM	**X**	**X**	---	ANAB
Dioxane, 1,4-	123-91-1	8270C-SIM	**X**	**X**	---	ANAB
Dioxane, 1,4-	123-91-1	8270D-SIM	**X**	**X**	---	ANAB
Endosulfan (Endosulfan I + Endosulfan II)	115-29-7	608	---	**X**	---	ANAB
Endosulfan (Endosulfan I + Endosulfan II)	115-29-7	8081A	**X**	**X**	---	ANAB
Endosulfan (Endosulfan I + Endosulfan II)	115-29-7	8081B	**X**	**X**	---	ANAB
Endosulfan I	959-98-8	608	---	**X**	---	ANAB
Endosulfan II	33213-65-9	608	---	**X**	---	ANAB
Endosulfan sulfate	1031-07-8	608	---	**X**	---	ANAB
Endrin	72-20-8	608	---	**X**	---	ANAB
Endrin	72-20-8	8081A	**X**	**X**	---	ANAB
Endrin	72-20-8	8081B	**X**	**X**	---	ANAB
Ethyl Chloride	75-00-3	8260B	**X**	**X**	---	ANAB
Ethyl Chloride	75-00-3	8260C	**X**	**X**	---	ANAB
Ethylbenzene	100-41-4	624	---	**X**	---	ANAB
Ethylbenzene	100-41-4	8260B	**X**	**X**	---	ANAB
Ethylbenzene	100-41-4	8260C	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	625	---	**X**	---	ANAB
Fluoranthene	206-44-0	8270C	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Fluoranthene	206-44-0	8270C-SIM	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	8270D	**X**	**X**	---	ANAB
Fluoranthene	206-44-0	8270D-SIM	**X**	**X**	---	ANAB
Fluorene	86-73-7	625	---	**X**	---	ANAB
Fluorene	86-73-7	8270C	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270C-SIM	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270D	**X**	**X**	---	ANAB
Fluorene	86-73-7	8270D-SIM	**X**	**X**	---	ANAB
Heptachlor	76-44-8	608	---	**X**	---	ANAB
Heptachlor	76-44-8	8081A	**X**	**X**	---	ANAB
Heptachlor	76-44-8	8081B	**X**	**X**	---	ANAB
Heptachlor Epoxide	1024-57-3	608	---	**X**	---	ANAB
Heptachlor Epoxide	1024-57-3	8081A	**X**	**X**	---	ANAB
Heptachlor Epoxide	1024-57-3	8081B	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	625	---	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270C	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270C-SIM	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270D	**X**	**X**	---	ANAB
Hexachlorobenzene	118-74-1	8270D-SIM	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	624	---	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	625	---	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260B	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260B-SIM	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260C	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8260C-SIM	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8270C	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8270C-SIM	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Hexachlorobutadiene	87-68-3	8270D	**X**	**X**	---	ANAB
Hexachlorobutadiene	87-68-3	8270D-SIM	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	608	---	**X**	---	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Alpha- (α-BHC)	319-84-6	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	608	---	**X**	---	ANAB
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Beta- (β-BHC)	319-85-7	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Delta- (δ-BHC)	319-86-8	608	---	**X**	---	ANAB
Hexachlorocyclohexane, Delta- (δ-BHC)	319-86-8	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Delta- (δ-BHC)	319-86-8	8081B	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	608	---	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	8081A	**X**	**X**	---	ANAB
Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	8081B	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270C	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270C-SIM	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270D	**X**	**X**	---	ANAB
Hexachlorocyclopentadiene	77-47-4	8270D-SIM	**X**	**X**	---	ANAB
Hexachloroethane	67-72-1	8270C	**X**	**X**	---	ANAB
Hexachloroethane	67-72-1	8270C-SIM	**X**	**X**	---	ANAB
Hexachloroethane	67-72-1	8270D	**X**	**X**	---	ANAB
Hexachloroethane	67-72-1	8270D-SIM	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260B	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260B-SIM	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260C	**X**	**X**	---	ANAB
Hexanone, 2-	591-78-6	8260C-SIM	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Indeno[1,2,3-cd]pyrene	193-39-5	625	---	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270C	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270C-SIM	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270D	**X**	**X**	---	ANAB
Indeno[1,2,3-cd]pyrene	193-39-5	8270D-SIM	**X**	**X**	---	ANAB
Isophorone	78-59-1	625	---	**X**	---	ANAB
Isophorone	78-59-1	8270C	**X**	**X**	---	ANAB
Isophorone	78-59-1	8270D	**X**	**X**	---	ANAB
Isopropanol	67-63-0	8260B-SIM	**X**	**X**	---	ANAB
Isopropanol	67-63-0	8260C-SIM	**X**	**X**	---	ORELAP
Lead, Total	7439-92-1	200.7	---	**X**	---	ANAB
Lead, Total	7439-92-1	200.8	---	**X**	---	ANAB
Lead, Total	7439-92-1	6010B	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6010C	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6020A	**X**	**X**	---	ANAB
Lead, Total	7439-92-1	6020B	**X**	**X**	---	ANAB
Mercury (elemental)	7439-97-6	7470A	---	**X**	---	ANAB
Mercury (elemental)	7439-97-6	7471A	**X**	---	---	ANAB
Methoxychlor	72-43-5	608	---	**X**	---	ANAB
Methoxychlor	72-43-5	8081A	**X**	**X**	---	ANAB
Methoxychlor	72-43-5	8081B	**X**	**X**	---	ANAB
Methyl Ethyl Ketone (2-Butanone)	78-93-3	8260B	**X**	**X**	---	ANAB
Methyl Ethyl Ketone (2-Butanone)	78-93-3	8260C	**X**	**X**	---	ANAB
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	624	---	**X**	---	ANAB
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	8260B	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	8260C	**X**	**X**	---	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	624	---	**X**	---	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	8260B	**X**	**X**	---	ANAB
Methyl tert-Butyl Ether (MTBE)	1634-04-4	8260C	**X**	**X**	---	ANAB
Methylene Chloride	75-09-2	624	---	**X**	---	ANAB
Methylene Chloride	75-09-2	8260B	**X**	**X**	---	ANAB
Methylene Chloride	75-09-2	8260C	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270C	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270C-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270D	**X**	**X**	---	ANAB
Methylnaphthalene, 1-	90-12-0	8270D-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270C	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270C-SIM	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270D	**X**	**X**	---	ANAB
Methylnaphthalene, 2-	91-57-6	8270D-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	624	---	**X**	---	ANAB
Naphthalene	91-20-3	625	---	**X**	---	ANAB
Naphthalene	91-20-3	8260B	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8260B-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8260C	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8260C-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270C	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270C-SIM	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270D	**X**	**X**	---	ANAB
Naphthalene	91-20-3	8270D-SIM	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	200.7	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Nickel, Total	7440-02-0	200.8	---	**X**	---	ANAB
Nickel, Total	7440-02-0	6010B	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	6010C	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	6020A	**X**	**X**	---	ANAB
Nickel, Total	7440-02-0	6020B	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	625	---	**X**	---	ANAB
Nitrobenzene	98-95-3	8270C	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8270C-SIM	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8270D	**X**	**X**	---	ANAB
Nitrobenzene	98-95-3	8270D-SIM	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	625	---	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270C	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270C-SIM	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270D	**X**	**X**	---	ANAB
Nitrosodimethylamine, N-	62-75-9	8270D-SIM	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	625	---	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270C	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270C-SIM	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270D	**X**	**X**	---	ANAB
Nitroso-di-N-propylamine, N-	621-64-7	8270D-SIM	**X**	**X**	---	ANAB
Nitrosodiphenylamine, N-	86-30-6	625	---	**X**	---	ANAB
Nitrosodiphenylamine, N-	86-30-6	8270C	**X**	**X**	---	ANAB
Nitrosodiphenylamine, N-	86-30-6	8270D	**X**	**X**	---	ANAB
Octyl Phthalate, di-N-	117-84-0	8270C	**X**	**X**	---	ANAB
Octyl Phthalate, di-N-	117-84-0	8270D	**X**	**X**	---	ANAB
PCB - Aroclor-1016	12674-11-2	608	---	**X**	---	ANAB
PCB - Aroclor-1016	12674-11-2	8082A	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
PCB - Aroclor-1221	11104-28-2	608	---	**X**	---	ANAB
PCB - Aroclor-1221	11104-28-2	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1232	11141-16-5	608	---	**X**	---	ANAB
PCB - Aroclor-1232	11141-16-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1242	53469-21-9	608	---	**X**	---	ANAB
PCB - Aroclor-1242	53469-21-9	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1248	12672-29-6	608	---	**X**	---	ANAB
PCB - Aroclor-1248	12672-29-6	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1254	11097-69-1	608	---	**X**	---	ANAB
PCB - Aroclor-1254	11097-69-1	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1260	11096-82-5	608	---	**X**	---	ANAB
PCB - Aroclor-1260	11096-82-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1262	37324-23-5	8082A	**X**	**X**	---	ANAB
PCB - Aroclor-1268	11100-14-4	8082A	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	625	---	**X**	---	ANAB
Pentachlorophenol	87-86-5	8151A	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270C	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270C-SIM	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270D	**X**	**X**	---	ANAB
Pentachlorophenol	87-86-5	8270D-SIM	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	625	---	**X**	---	ANAB
Phenanthrene	85-01-8	8270C	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270C-SIM	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270D	**X**	**X**	---	ANAB
Phenanthrene	85-01-8	8270D-SIM	**X**	**X**	---	ANAB
Phenol	108-95-2	625	---	**X**	---	ANAB
Phenol	108-95-2	8270C	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Phenol	108-95-2	8270D	**X**	**X**	---	ANAB
Propyl benzene	103-65-1	624	---	**X**	---	ANAB
Propyl benzene	103-65-1	8260B	**X**	**X**	---	ANAB
Propyl benzene	103-65-1	8260C	**X**	**X**	---	ANAB
Pyrene	129-00-0	625	---	**X**	---	ANAB
Pyrene	129-00-0	8270C	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270C-SIM	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270D	**X**	**X**	---	ANAB
Pyrene	129-00-0	8270D-SIM	**X**	**X**	---	ANAB
Selenium	7782-49-2	6010B	**X**	**X**	---	ANAB
Selenium	7782-49-2	6010C	**X**	**X**	---	ANAB
Selenium	7782-49-2	6020A	**X**	**X**	---	ANAB
Selenium	7782-49-2	6020B	**X**	**X**	---	ANAB
Silver	7440-22-4	6010B	**X**	**X**	---	ANAB
Silver	7440-22-4	6010C	**X**	**X**	---	ANAB
Silver	7440-22-4	6020A	**X**	**X**	---	ANAB
Silver	7440-22-4	6020B	**X**	**X**	---	ANAB
Styrene	100-42-5	624	---	**X**	---	ANAB
Styrene	100-42-5	8260B	**X**	**X**	---	ANAB
Styrene	100-42-5	8260C	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260B	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260B-SIM	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260C	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,1,2-	630-20-6	8260C-SIM	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260B	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260B-SIM	**X**	**X**	---	ANAB
Tetrachloroethane, 1,1,2,2-	79-34-5	8260C	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Tetrachloroethane, 1,1,2,2-	79-34-5	8260C-SIM	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	624	---	**X**	---	ANAB
Tetrachloroethylene	127-18-4	8260B	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	8260B-SIM	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	8260C	**X**	**X**	---	ANAB
Tetrachloroethylene	127-18-4	8260C-SIM	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	200.7	---	**X**	---	ANAB
Thallium, Total	7440-28-0	200.8	---	**X**	---	ANAB
Thallium, Total	7440-28-0	6010B	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6010C	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6020A	**X**	**X**	---	ANAB
Thallium, Total	7440-28-0	6020B	**X**	**X**	---	ANAB
Toluene	108-88-3	624	---	**X**	---	ANAB
Toluene	108-88-3	8260B	**X**	**X**	---	ANAB
Toluene	108-88-3	8260C	**X**	**X**	---	ANAB
Toxaphene	8001-35-2	608	---	**X**	---	ANAB
Toxaphene	8001-35-2	8081A	**X**	**X**	---	ANAB
Toxaphene	8001-35-2	8081B	**X**	**X**	---	ANAB
Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	76-13-1	8260B	**X**	**X**	---	ORELAP
Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	76-13-1	8260C	**X**	**X**	---	ORELAP
Trichlorobenzene, 1,2,3-	87-61-6	624	---	**X**	---	ANAB
Trichlorobenzene, 1,2,3-	87-61-6	8260B	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,3-	87-61-6	8260C	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	624	---	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	625	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Trichlorobenzene, 1,2,4-	120-82-1	8260B	**X**	**X**	---	ANAB
Trichlorobenzene, 1,2,4-	120-82-1	8260C	**X**	**X**	---	ANAB
Trichloroethane, 1,1,1-	71-55-6	624	---	**X**	---	ANAB
Trichloroethane, 1,1,1-	71-55-6	8260B	**X**	**X**	---	ANAB
Trichloroethane, 1,1,1-	71-55-6	8260C	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	624	---	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260B	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260B-SIM	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260C	**X**	**X**	---	ANAB
Trichloroethane, 1,1,2-	79-00-5	8260C-SIM	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	8260B	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	8260B-SIM	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	8260C	**X**	**X**	---	ANAB
Trichloroethylene	79-01-6	8260C-SIM	**X**	**X**	---	ANAB
Trichlorofluoromethane	75-69-4	624	---	**X**	---	ANAB
Trichlorofluoromethane	75-69-4	8260B	**X**	**X**	---	ANAB
Trichlorofluoromethane	75-69-4	8260C	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,5-	95-95-4	8270C	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,5-	95-95-4	8270D	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	625	---	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270C	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270C-SIM	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270D	**X**	**X**	---	ANAB
Trichlorophenol, 2,4,6-	88-06-2	8270D-SIM	**X**	**X**	---	ANAB
Trichlorophenoxyacetic Acid, 2,4,5- (2,4,5-T)	93-76-5	8151A	**X**	**X**	---	ANAB
Trichlorophenoxypropionic acid, 2,4,5-(2,4,5-TP)	93-72-1	8151A	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Trichloropropane, 1,2,3-	96-18-4	624	---	**X**	---	ANAB
Trichloropropane, 1,2,3-	96-18-4	8011	**X**	**X**	---	ANAB
Trichloropropane, 1,2,3-	96-18-4	8260B	**X**	**X**	---	ANAB
Trichloropropane, 1,2,3-	96-18-4	8260C	**X**	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	624	---	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	8260B	**X**	**X**	---	ANAB
Trimethylbenzene, 1,2,4-	95-63-6	8260C	**X**	**X**	---	ANAB
Trimethylbenzene, 1,3,5-	108-67-8	624	---	**X**	---	ANAB
Trimethylbenzene, 1,3,5-	108-67-8	8260B	**X**	**X**	---	ANAB
Trimethylbenzene, 1,3,5-	108-67-8	8260C	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	200.7	---	**X**	---	ANAB
Vanadium, Total	7440-62-2	6010B	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6010C	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6020A	**X**	**X**	---	ANAB
Vanadium, Total	7440-62-2	6020B	**X**	**X**	---	ANAB
Vinyl Acetate	108-05-4	624	---	**X**	---	ANAB
Vinyl Acetate	108-05-4	8260B	**X**	**X**	---	ANAB
Vinyl Acetate	108-05-4	8260C	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	624	---	**X**	---	ANAB
Vinyl Chloride	75-01-4	8260B	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	8260B-SIM	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	8260C	**X**	**X**	---	ANAB
Vinyl Chloride	75-01-4	8260C-SIM	**X**	**X**	---	ANAB
Xylene, m+p -	-	624	---	**X**	---	ANAB
Xylene, m+p -	-	8260B	**X**	**X**	---	ANAB
Xylene, m+p -	-	8260C	**X**	**X**	---	ANAB
Xylene, o-	95-47-6	624	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Xylene, o-	95-47-6	8260B	**X**	**X**	---	ANAB
Xylene, o-	95-47-6	8260C	**X**	**X**	---	ANAB
Xylene, Total	1330-20-7	624	---	**X**	---	ORELAP
Xylene, Total	1330-20-7	8260B	**X**	**X**	---	ORELAP
Xylene, Total	1330-20-7	8260C	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	200.7	---	**X**	---	ANAB
Zinc, Total	7440-66-6	200.8	---	**X**	---	ANAB
Zinc, Total	7440-66-6	6010B	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6010C	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6020A	**X**	**X**	---	ANAB
Zinc, Total	7440-66-6	6020B	**X**	**X**	---	ANAB
Total Organic Carbon	N/A	9060	**X**	**X**	---	ANAB
Total Organic Carbon	N/A	Lloyd Kahn	---	---	---	---
Total Organic Carbon	N/A	SM 5310 B	---	**X**	---	ANAB
Total Organic Carbon	N/A	Walkley Black	---	---	---	---
Gasoline Range Organics (C6 – C10)	N/A	AK 101	**X**	**X**	---	ANAB
Diesel Range Organics (C10 – C25)	N/A	AK 102	**X**	**X**	---	ANAB
Residual Range Organics (C25 – C36)	N/A	AK 103	**X**	**X**	---	ANAB
Aliphatic Petroleum Hydrocarbons (C10 – C12)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C12 – C16)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C16 – C21)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C21 – C34)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Aliphatic Petroleum Hydrocarbons (C8 – C10)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C10 – C12)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C12 – C16)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C16 – C21)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C21 – C34)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C8 – C10)	N/A	NWTPH EPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C10 – C12)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C5 – C6)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C6 – C8)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aliphatic Petroleum Hydrocarbons (C8 – C10)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C10 – C12)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C12 – C13)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
Aromatic Petroleum Hydrocarbons (C8 – C10)	N/A	NWTPH VPH	**X**	**X**	---	ORELAP
TCLP Extraction	N/A	1311	**X**	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
SPLP	N/A	1312	**X**	**X**	---	ORELAP
Acid Digestion for Metals Analysis	N/A	3010A	---	---	---	---
Microwave Assisted Acid Digestion	N/A	3015A	---	---	---	---
Acid Digestion	N/A	3050B	**X**	**X**	---	ANAB
Microwave Assisted Acid Digestion	N/A	3050B	---	---	---	---
Microwave Assisted Acid Digestion	N/A	3051A	---	---	---	---
Alkaline Digestion	N/A	3060A	---	---	---	---
Separatory Funnel Extraction	N/A	3510C	---	**X**	---	ANAB
Liquid-Liquid Extraction	N/A	3520C	---	**X**	---	ANAB
SPE extraction for explosives	N/A	3535A	---	---	---	---
Soxhlet Extraction	N/A	3540C	---	---	---	---
Pressurized Fluid Extraction (PFE)	N/A	3545	---	---	---	---
Microwave Extraction	N/A	3546	**X**	---	---	ANAB
Ultrasonic Extraction	N/A	3550B	**X**	**X**	---	ANAB
Ultrasonic Extraction	N/A	3550C	---	---	---	---
Alumina Cleanup	N/A	3610B	---	---	---	---
Florisil Cleanup	N/A	3620B	**X**	**X**	---	ANAB
Florisil Cleanup	N/A	3620C	---	---	---	---
Acid Base Partition Cleanup	N/A	3650B	---	---	---	---
Sulfur cleanup	N/A	3660B	**X**	**X**	---	ANAB
Sulfuric Acid/Permanganate Cleanup	N/A	3665A	**X**	**X**	---	ANAB
Purge and Trap	N/A	5030B	**X**	**X**	---	ANAB
Purge and Trap	N/A	5030C	---	---	---	---
Closed System Purge and Trap	N/A	5035	---	---	---	---
Closed System Purge and Trap	N/A	5035A	**X**	**X**	---	ANAB
Closed-System Purge and Trap	N/A	5035B	---	---	---	---
Mercury Digestion	N/A	7470A	---	**X**	---	ANAB

Scope of Approval – X indicates approved methods

Hazardous Substance	CAS Number	Analysis Method	Sample Matrix			Accrediting Body
			Soil	Water	Air	
Mercury Digestion	N/A	7470B	---	---	---	---
Mercury Digestion	N/A	7471A	**X**	---	---	ANAB
Incremental Sampling	N/A	8330B	---	---	---	---
Acid Digestion for Metals	N/A	3005A	---	**X**	---	ANAB

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Toxicity Characteristic Leaching Procedure (TCLP, Method 1311) and Synthetic Precipitation Leaching Procedure (SPLP, Method 1312)

Approvals

Signatures on File	
Stan Palmquist Date	Manjit Nijjar Date
Inorganic Department Manager	Health & Safety Coordinator
Terri Torres Date	Dennis Bean Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 Both TCLP and SPLP are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP or SPLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP or SPLP 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.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

1.5 On occasion, clients may request slight modifications to this SOP. These modifications are addressed on a case by case basis with the supporting demonstration of sensitivity and accuracy (e.g. MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into project special instructions (e.g. Quality Assurance Project Plans), authorized by the laboratory, and mentioned in the report narrative.

2.0 Method Summary

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.7 µm glass fiber filter, is defined as the TCLP extract.

2.2 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 µm to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.3 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A ZHE vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through the appropriate glass fiber filter.

2.4 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together.

2.5 If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Definitions

3.1 Batch: A group of 20 or less samples prepared and/or processed together within the same shift using the same reagents. Each batch must contain a minimum QC of one method blank.

3.2 Leachate: The TCLP or SPLP solution generated after the solids are tumbled with the appropriate fluid.

3.3 Liquid Phase: The liquid fraction or filtrate of a sample that passes through a 0.6 to 0.8μm fiber filter.

3.4 Final Leachate: The final solution generated from this procedure – either a leachate or a leachate combined with a filtrate.

3.5 Percent Solids: The fraction of a sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

3.6 Solid phase: The material in the filter holder that did not pass through a 0.6 to 0.8μm fiber filter.

4.0 Interferences and Comments

4.1 Oily samples may present unusual filtration and drying problems. For example, the oily sample may pre-maturely clog the filter used in the percent wet solids determination, causing a high-biased result for percent wet solids. As recommended by EPA (see Figure 3 in Appendix B), oily samples will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. For oily samples requiring metals analysis, a suitable sub-lab will be procured. For oily wastes requiring semi-volatile organic analysis, the sample should be logged into the LIMS as a solid matrix for waste dilution extraction. For oily samples requiring volatile organic analysis, the sample should be logged into the LIMS as an aqueous matrix for analysis.

4.2 Samples containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the sample exceeds TCLP limits.

4.3 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.4 Over exposure of the sample to the environment will result in the loss of volatile components.

4.5 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.6 The laboratory analyst will perform the method in accordance with this SOP. The analyst will resolve non-conformances in methods and data, either individually or with the assistance of the Department Supervisor or Operations Manager. Deviations from this SOP must be documented. Bench sheets and raw data must capture information related

to a deviation. The laboratory analyst or supervisor will report deviations or non-conforming events to the Operations, Project and/or QA Manager via a non-conformance report.

4.7 The Department Supervisor, Operations Manager, and/or QA Manager will assist the laboratory analyst in resolving non-conformances.

4.8 The Department Supervisor will review and approve bench sheets and notebooks.

4.9 The QA Manager shall monitor adherence to this SOP through annual audits and non-conformance reports.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Specific safety concerns or requirements as they related to this procedure include.

5.1 The importation and movement of untreated soil is considered by APHIS to be an extremely high-risk activity, because it can readily provide a pathway for the introduction of a variety of dangerous organisms into the United States. In addition to containing high concentrations of hazardous and or toxic compounds, regulated soils and effluents may contain plant, animal or human disease agents.

5.2 Spills of regulated soil and water residue are to be handled as follows:

Spray generously with bleach, before attempting to clean the spill up to prevent the release of airborne contaminants. The area should be cleaned with bleach again after the spill has been removed. Place all clean up material, spilled sample and PPE into a sealable container for autoclaving.

5.3 The filtration of all TCLP samples and extracts are performed in a hood or well-ventilated area. All excess sample/ extract waste is discarded into TCLP (Toxicity Characteristic Leaching Procedure) labeled waste containers for hazardous waste disposal.

5.4 Unknown samples may contain high concentrations of toxic volatile compounds. Sample containers are to be opened in a hood and handled with gloves to prevent exposure.

5.5 Glassware should be inspected for chips or cracks before use. Chipped/broken glassware that poses a safety hazard must be removed from service and repaired or discarded.

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table**

contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS. Electronic copies of SDS can be located on the EH&S webpage of Oasis.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Documentation and Maintenance

6.1 All maintenance performed on any TCLP/SPLP agitation apparatus, ZHE extraction vessel, or Millipore filtration apparatus must be documented in a TCLP Maintenance logbook.

6.2 ZHE Extraction Vessel Pressure Checks

6.2.1 Each ZHE extraction vessel undergoes a one-hour pressure check before each use to identify any major pressure-related malfunctions **prior** to extraction.

6.2.1.1 Fully assemble and pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer must be contacted**.** Recheck the pressure every hour for four hours when doing the monthly pressure check. Log all results in the ZHE pressure check log.

6.2.2 A 4-hour pressure check on each ZHE extraction vessel must be performed and documented monthly. This is done to check for any slow leaks that pass inspection otherwise.

6.2.3 Refer to Appendix A for a template of the ZHE Pressure Check Log.

6.2.4 Any ZHE body that fails the pressure check must be placed on a cart with an "out of service" marker attached to prevent intermingling with acceptable ZHE vessels.

6.3 All TCLP/SPLP agitation devices must be checked monthly, under a full load, to insure they rotate at 30±2 rpm. Document their rotation in the TCLP Tumbler Check Log (see Appendix B).

6.4 *As needed the* ZHE metal filter screens are sent through the sonic bath for 15 minutes (in enough MeOH or DIW to cover all the screens) after each use to shake out any sample that may be clogged in the pores of the filters.

6.5 ZHE O-rings should be inspected prior to each use for tears and contamination, and their replacement documented in the ZHE Pressure Check Logbook.

6.6 All extraction vessel Blanks must be documented in the appropriate TCLP/ZHE Logs. (Appendices D & E)

6.7 The ambient temperature must be recorded on the appropriate log sheet. If the corrected temperature range during an extraction falls outside the method-specified range of 23 ± 2°C, initiate a NCM as prescribed in TA-QA-610.

7.0 Equipment and Supplies

7.1 Agitation apparatus – Tumbler #1 and #2-Environmental Express Multi-Place, 10 positions, capable of maintaining a tumbling speed of 30± 2 rpm (or equivalent device).

Extraction Vessels

7.1.1 Millipore ZHE Extraction system, Zero-Headspace Extraction Vessels (ZHE), uniquely numbered.

Note: 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. For the ZHE to be acceptable for use, the piston within the ZHE must be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device must be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer must be contacted.

7.1.2 ESS 128oz or 68oz pre-cleaned 1 gallon disposable poly bottles-one time use only

7.1.3 ESS 128oz or 68oz pre-cleaned 1 gallon disposable borosilicate glass bottles-one time use only.

Note: When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. Plastic bottles, other than polytetrafluoroethylene (PTFE), shall not be used if organics are to be investigated.

7.2 Filtration Devices

7.2.1 Positive pressure filtration device -- Millipore Hazardous Waste Filtration System.

7.2.2 ZHE Filters: TCLP Glass Fiber 0.7μm, 90mm. Whatman cat # 1810-090

7.2.3 TCLP Filters: Borosilicate Acid Washed Glass Fiber Filters, pore size 0.6 to 0.8μm, 142 mm. Whatman acid washed low level cat # 1810-142

Note: Glass fiber filters are fragile and should be handled with care.

7.3 pH Meters: Denver Instruments pH meter (or equivalent), resolution to 0.01 pH units.

7.4.1 Calibration of the Denver Instruments pH meter:

7.4.1.1 Turn-on the unit.

7.4.1.2 Dip electrode ½" to 1" into buffer solution of pH = *2.00*

7.4.1.3 Press the standardize button to enter Calibration mode. When the 'S' appears on the screen, remove the probe, rinse, and repeat with buffer solution pH 7.00 and 10.00.

7.4.1.4 After reading buffer 10, the calculated slope will appear on the screen. Document this in the TCLP logbook and verify it is within acceptance criteria before proceeding.

7.4.1.5 Calibration with a second source pH = 7.00 is required also and is recorded in the TCLP logbook and must fall with in 7.00 +/- .05.

7.4.1.6 If the probe does not calibrate properly you must perform maintenance and repeat the process.

7.4.2 pH testing with the Denver Instruments pH meter.

7.4.2.1 Turn on the unit

7.4.2.2 Dip the electrode ½" to 1" into the test solution. Stir once and let the reading stabilize. Record the pH to 0.01 pH unit in TCLP logbook or in the TALS worksheet.

7.4 FT Variable Speed Metering Pump (ZHE pump) for transferring ZHE extraction fluid.

7.5 Top-Loading Balance: capable of reading to 0.1g.

7.6 Beaker, plastic, 200 mL

7.7 500 mL and 2000 mL Graduated cylinders

7.8 Watch glass, appropriate diameter to cover beaker.

7.9 Hot plate.

7.10 Wood spatulas or tongue depressors (or equivalent)

7.11 Magnetic stir bar.

7.12 Teflon Boiling Chips

7.13 Teflon Tape

7.14 Centrifuge, Beckman TJ-6 or Equivalent

7.15 Narrow range pH strips- pH 0-6, pH 5-10, pH 7.5-14

8.0 Reagents and Standards

All reagents and standards used in this procedure must conform to the requirements specified in TA-QA-0619. Preparation of reagents and standards will be documented in the LIMS system and a label will be generated to include, as appropriate: the name and expected concentration of the material; the generated LIMS ID number; the date of preparation; the expiration date; the analyst's initials.

8.1 pH Buffers for meter calibration, pH 2, pH 7 and pH 10.

8.2 Deionized Water.

8.3 Nitric acid (HNO_3).

8.4 2% Nitric acid solution for rinsing TCLP filter.

8.4.1 In a 1L poly wash bottle add between 500 and 800 mls of DI water. Add 20mls of Nitric acid, top off to 1000mls with DI water, and mix.

8.5 Glacial acetic acid (CH3COOH)

8.6 Sodium Hydroxide Pellets (NaOH).

8.7 *Reagents be used only if **not** making TCLP fluid #1 from concentrate*

8.7.1 1N Sodium hydroxide (NaOH).

8.7.1.1 In a 1 liter volumetric flask, add 250 mLs of reagent water. Dissolve 40g of NaOH pellets into this, adding more reagent water as it slowly dissolves. Dilute to a final volume of 1 liter with reagent water. Document in the reagent module in TALS, generate a label and place on the bottle.

8.8 Sulfuric acid/nitric acid (60/40 weight percent) mixture

8.8.1 The amount specified to be used in the extraction fluid preparation is so minimal that it is useful to create a 100X dilution of the mix to increase the measuring accuracy. Amounts are not specified in the EPA method. Only the final pH of the extraction fluid is specified. Therefore, these amounts may need to be adjusted to ensure proper pH.

8.9 Extraction fluids.

8.9.1 The extraction fluids are prepared in-house by the technician using the procedure described below.

8.9.2 TCLP Fluid Mix #1 (Concentrate)

8.9.2.1 In a carboy, dissolve approximately 1040 g NaOH pellets into 10 L reagent water.

8.9.2.2 Add approximately 2.3 L glacial acetic acid to carboy.

8.9.2.3 Dilute to a volume of 20 liters. When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

8.9.3 TCLP Fluid # 1 for TCLP and ZHE

from concentrate

8.9.3.1 Add 1 L TCLP Fluid Mix #1 (Concentrate) to 19 L reagent water in a carboy.

8.9.3.2 When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

8.9.3.3 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.3.3.1 If the pH isn't within this range, a new solution must be prepared.

8.9.4 TCLP Fluid Mix #1 (Not from concentrate)

8.9.4.1 Add approximately 115 mL glacial acetic acid to 15 L of reagent water in a carboy.

8.9.4.2 Add approximately 1300 mL of 1N NaOH.

8.9.4.3 Dilute to a volume of 20 liters. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

8.9.4.4 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.3.7.1 If the pH isn't within this range, a new solution must be prepared.

8.9.5 TCLP Fluid # 2

8.9.5.1 Add 5.7 mL glacial acetic acid to 500 ml reagent water.

8.9.5.2 Bring to a final volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

8.9.5.3 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.9.3.3.1 If the pH isn't within this range, a new solution must be prepared.

8.9.6 SPLP Fluid #1

8.9.6.1 Add 29.25 uL of the 60/40 sulfuric acid/nitric acid mix (8.8) to 18 liters of reagent water in a carboy. If using the 100X acid dilution then add 2.925 mLs of acid to 18 liters of DI water.

8.9.6.2 Dilute to a volume of 20 liters.

8.9.6.3 When correctly prepared, the final pH of this fluid should be 4.20 ± 0.05.

8.9.6.4 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.9.5.4.1 If the pH isn't within this range, a new solution must be prepared.

8.9.7 SPLP Fluid #2

8.9.7.1 Add 6.75 uL of the 60/40 sulfuric acid/nitric acid mix (8.8) to 18 liters of reagent water in a carboy. If using the 100X acid dilution then add 0.675 mLs of acid to 18 liters of DI water.

8.9.7.2 Dilute to a volume of 20 liters.

8.9.7.3 When correctly prepared, the final pH of this fluid should be 5.00 ± 0.05.

8.9.7.4 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.6.4.1 If the pH isn't within this range, a new solution must be prepared.

8.9.8 SPLP Fluid #3

8.9.8.1 Dionized water

8.10 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

Note: These extraction fluids must be documented in the TALS and monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid must be discarded and fresh extraction fluid prepared.

9.0 Sample Collection, Preservation, Shipment and Storage

9.1 Preservatives shall not be added to samples before extraction.

9.2 Samples are refrigerated at 4°C unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) is extracted.

9.3 When the waste is to be evaluated for volatile analytes, care must be taken to minimize the loss of volatiles. Samples are stored at 4°C to prevent the loss of volatile analytes. Samples are opened only immediately prior to extraction.

9.4 Hold times and minimum sample amounts are specified in Table 1.

9.5 TCLP and SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for volatile organic analyte determinations are not allowed to come into contact with the atmosphere any longer than is absolutely necessary (i.e., no headspace) to prevent losses. Extracts or portions of extracts for metallic analyte determinations are acidified with nitric acid to a pH < 2, unless precipitation occurs. The metals prep group will adjust the pH so they can add their spike solutions prior to the pH adjustment. Extracts are preserved for other analytes according to the guidance given in the individual analysis methods.

10.0 Quality Control

10.1 Method Blanks

10.1.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. If both extraction fluids are used in a batch, two method blanks need to be prepared. One for each extraction fluid.

10.1.2 The method blank should contain no analyte(s) of interest at concentrations greater that the method reporting limit.

10.1.3 If method blank failure occurs, a Non-Conformance Report must be initiated, and all samples associated with the analytical batch must be reprocessed.

10.2 Samples must undergo TCLP or SPLP extraction within the time periods outlined in Table 01. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

10.3 A matrix spike and matrix spike dup must be performed for each waste type unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. This is performed post TCLP as part of the Organic extraction or Inorganic digestion process.

10.3.1 Matrix spike solutions are to be added after filtration of the TCLP or SPLP extract and before preservation.

10.4 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

Note: An LCS/LCSD set is created on the TCLP/SPLP/ZHE prep batch but are only there for use when preparing batches for post TCLP processes. Each department uses a portion of the MB extract to create the LCS and LCSD, if necessary. The information in the TCLP prep batch for the LCS and LCSD should be documented identical to the MB.

11.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of the department manager to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

Document the results for each sample preparation procedure in TALS (worksheet)

NOTE: SAMPLE PREPARATION
The TCLP extraction depends on the analysis and matrix of each sample. Before the extraction process can be started, the following preliminary evaluations must take place:

- 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 nonvolatile TCLP extraction of the waste.

A **minimum** 100 g aliquot of waste is required to perform these preliminary evaluations. This aliquot may not actually undergo TCLP extraction.

11.1 Preliminary Evaluations for TCLP and SPLP

Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014.

11.1.1 Percent Solids Determination

Percent solids are defined as that fraction of a waste sample from which no liquid may be forced out by an applied pressure.

11.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration at 50 PSI for 2 minutes, it is 100% solid. Proceed to section 11.1.2 to determine whether the solid material requires particle size reduction.

11.1.1.2 If the sample is liquid and is obviously <0.5% solids (for instance, a clear sample with no particles present in the sample), then it is 0% solids. If it contains particulates or is cloudy, then a percent solids determination must be performed as described in 11.1.1.3.

Note: In practice, the analyst has been contacting the Project Manager regarding multiphasic samples. If the client requests that each phase be treated separately, percent solids determination is performed on the individual phases. Otherwise, the phases (usually a settled solid layer, and a liquid layer) are homogenized prior to the determination.

11.1.1.3 If the sample is multiphasic, each layer must be separated and a percent solids determination must be performed on each layer.

The sample, a receiving container, and a filter paper are needed for this evaluation.

11.1.1.3.1 Pre-weigh and record each of the individual weights to the nearest tenth of a gram for the sample, the filter, and the container that will receive the filtrate.

11.1.1.3.2 Homogenize the sample with a wood spatula. Weigh the sample of the waste (100 g minimum), and record the weight to the nearest tenth of a gram.

11.1.1.3.3 Allow slurries to stand to permit the solid phase to settle.

Note: If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

11.1.1.3.4 Quantitatively transfer the waste sample into the filtration device (liquid and solid phases). Spread the waste sample evenly over the surface of the filter.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight to determine the weight of the waste sample that will be filtered.

11.1.1.3.5 Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

11.1.1.3.6 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined above, this material may 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**.

11.1.1.3.7 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container.

11.1.1.3.8 Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample.

11.1.1.3.9 Record the weight of the liquid and solid phases to the nearest tenth of a gram on the bench sheet.

11.1.1.3.10 Calculate the percent solids as follows:

$$\% \text{ Solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

11.1.1.3.11 If the percent solids are less than 0.5%, then the filtrate is the sample. Proceed to section 11.2.1.3.

11.1.1.3.12 If the percent solids are greater than or equal to 0.5% and it's noticed that a small amount of the filtrate is entrained in the wetting of the filter, determine the percent dry solids.

11.1.1.3.12.1 Remove the solid phase and filter from the filtration apparatus.

11.1.1.3.12.2 Dry the filter and solid phase at 100 ± 20°C. Weigh the filter/solid phase every hour until two successive weights yield the same value within ± 1%.

Note: Use the properly-vented Percent Solids oven for drying these samples.

11.1.1.3.12.3 Record the final weight.

11.1.1.3.12.4 Calculate the percent dry solids as follows:

$$\% \text{ Dry Solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste}} * 100$$

11.1.1.3.12.5 If the percent dry solids is <0.5%, then the waste phase is 0% solids.

11.1.1.3.12.6 If the percent dry solids is ≥ to 0.5%, then proceed to section 11.1.2, Particle Size Reduction.

11.1.1.3.13 If the percent solids are greater than or equal to 0.5%, then proceed to section 11.1.2 to determine whether the solid material requires particle size reduction.

Note: If organic or volatile compounds are to be analyzed, then you must extract/ filter a fresh portion of the sample.

11.1.2 Particle Size Reduction for TCLP and SPLP

This section determines whether the waste requires particle size reduction. This section also describes how to reduce particle size.

11.1.2.1 Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required unless:

11.1.2.1.1 The solid has a surface area per gram of material equal to or greater than 3.1 cm^2.

11.1.2.1.2 The solid is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve).

Note: Do not sieve the waste if volatiles are being analyzed because some of the volatiles may be lost.

11.1.2.2 If particle size reduction is required, then:

11.1.2.2.1 Prepare the solid portion of the waste for extraction by crushing, cutting, grinding, mashing, ripping, or tearing the waste to a surface area or particle size as described above. Care is taken to reduce contamination of samples by washing the tools used in particle size reduction with water and gentle soap, and rinsing 3 times with DI water.

11.1.2.2.2 If the solids are prepared for organic volatiles extraction, special precautions must be taken so that heat is not generated from the reducing process. The sample, and the tools to be used, should be refrigerated at 4˚ C prior to particle size reduction.

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. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

11.1.2.3 If particle size reduction is required, but not feasible with the available apparatus and equipment, notify the department supervisor and initiate a non-conformance report.

11.1.3 Extraction fluid determination for TCLP and SPLP

This section determines the appropriate TCLP extraction fluid.

11.1.3.1 If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the nonvolatile extraction as follows:

Note: TCLP extraction for volatile constituents (ZHE) uses only extraction fluid #1 and is prepared with water proven to be interference free.

Note: For Non-aqueous liquid samples, such as products, that need to go through the tumbling process, a pH test is not necessary.

11.1.3.1.1 Weigh out 5g of the sample into a 200 mL plastic beaker. Where there is no exemption for density or surface area the lab will take reasonable efforts to reduce the solid to a particle size of approximately 1 mm in diameter or less.

11.1.3.1.2 Add 96.5 mL of deionized water to the beaker, drop in a stir bar, cover with a watch glass, and stir vigorously on a magnetic stirrer for 5 minutes. Check the pH and if it is <5.0, use extraction fluid #1. Record the pH in the worksheet in TALS.

11.1.3.1.3 If the pH is >5.0, add 3.5 mL 1N HCl, stir on medium, cover with a watch glass, heat to 50°C ± 5°C on a hot plate. Hold at 50°C ± 5°C for 10 minutes ± 1 minute.

11.1.3.1.4 Remove the watch glass and let the solution cool to room temperature, (around ½ an hour), test and record the pH in TALS.

If the pH is <5.0, use TCLP Fluid #1. Record the lot number of the fluid in the worksheet in TALS.

11.1.3.1.5 If the pH is >5.0, use TCLP Fluid #2. Record the lot number of the fluid in the worksheet in TALS.

11.1.3.1.6 Record the pH to 0.01 pH unit that resulted from this procedure in the worksheet in TALS.

This section determines the appropriate SPLP extraction fluid

11.1.3.2 If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the nonvolatile extraction as follows:

Note: SPLP extraction for volatile constituents (ZHE) and cyanide uses only SPLP Fluid #3 (Purged reverse osmosis type III water).

11.1.3.2.1 SPLP Fluid #1 is used to determine the leachability of contaminants from soils originating from east of the Mississippi River, as well as the leachability of wastes and wastewaters.

11.1.3.2.2 SPLP Fluid #2 is used to determine the leachability of contaminants from soils originating from west of the Mississippi River.

List of General Analyses for TCLP and SPLP

Table 1 – TCLP and SPLP Method Specifications and Recommended Sample.

Analysis	Type extraction vessel	Where filtered	Type extract container	QC	Extract preservative	Hold time to TCLP	Hold time to extract	Mass solid phase (minimum # grams)	Vol. Final Extract (mLs)
Metals	1L HDPE Poly	Plunger Filters	500mL HDPE Poly or Evergreen tubes	Blank/BS/ MS/MSD every 20 or per batch	pH<2 HNO3 unless Sx precipitates	180 days. Mercury= 28 days	180 days. Mercury =28 days	100	20X mass of solid
Organics in general	2L Teflon or 68oz Teflon	Millipore	1L Amber glass or 40 mL glass VOA	Refer to specific method	Refer to specific method	Refer to specific method	Refer to specific method	Refer to specific method (Usually 100)	20X mass of solid, or refer to method for liquids
8270 C Semi-volatiles	2L Teflon or 68oz Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
Volatiles	ZHE extraction vessel	ZHE extraction vessel	5 mL gass-tight glass syringe	Blank/ 20	No headspace	14 days	14 days	25	20X mass of solid
8081A 8082B Pesticides/ PCB's	2L Teflon or 68oz Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8310 PAH	2L Teflon or 68oz Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8015 TPH-D	2L Teflon or 68oz Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8151 Herbicide	2L Teflon or 68oz Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid

Note: The TCLP and SPLP methods require 100 g minimum sample amount. If less than 100 g is used, a Non-Conformance Memo must be issued. ZHE samples use 25 g, and do not require a NCM unless less than 25 g is available.

11.2 TCLP and SPLP Non-Volatiles Extraction Procedure

NOTE: Document sample information in the TCLP/ SPLP Sample Log (see Appendices C & D).

11.2.1 Procedure by matrix

11.2.1.1 If the sample is determined to be 100% solid and the preparation steps in section 11.1 were completed, then weigh out a minimum of 100 g of

homogenized sample using a wood spatula or tongue depressor, making sure that there is an appropriate amount of sample to yield the amount of extract required by the specific analysis. Record the weight in the logbook to the nearest tenth of a gram. The ratio of extraction fluid added to 100% solid sample is 20 mLs to 1 g (20:1). In this non-volatile extraction procedure, the initial 100 g weighed out for the preliminary evaluation in section 11.1 may be used to extract the sample. Place the weighed sample and extraction fluid in the correct extraction vessel listed in Table-01. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. Go to Section 11.2.3 for Tumbling.

11.2.1.2 If the sample contains less than 100% solids, liquid/solid separation is required. For the non-volatile extraction procedure, the filtrate and solid portion/filter obtained in the percent solids determination (section 11.1.1) may be used. The liquid phase obtained during the percent solids determination is collected in an appropriate, labeled, pre-weighed container, re-weighed, and set aside. The weight of the filtrate and the solid portion/filter was recorded on the bench sheet to the nearest tenth of a gram (see section 11.1.1.3.9). The other preliminary evaluations identified in sections 11.1.2 and 11.1.3 were completed.

11.2.1.2.1 The solid portion/filter is placed in the correct extractor vessel. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. The weight of extraction fluid to be added is calculated according to the equation below:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

For example: 100 g of the sample was weighed out and the percent solids were calculated at 25%.

$$\text{Weight of extraction fluid (g)} = \frac{20 \times 25\% \text{ solids} \times 100 \text{ g total weight of waste filtered}}{100\%}$$

The weight of extraction fluid to add to the extraction vessel in this case is 500 g.

11.2.1.2.2 The solid portion will be tumbled as outlined in Section 11.2.3.

11.2.1.3 If the sample is determined to be <0.5% solids, then the filtrate obtained from the percent solids determination is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the analyses required of the TCLP extract.

Note: If the client has not provided sufficient sample (100 g minimum), then the Project Manager must be notified through a Non-Conformance Report. If sufficient sample cannot be obtained, then the data will be qualified. The qualifier will read, “Due to insufficient sample provided for analysis, a lesser aliquot was analyzed than the method-required 100 grams.”

Note: 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. Also, if you used a portion of your initial 100 g to perform a fluid determination, remember to take that portion into account during the rest of the procedure.

11.2.2 Method Blanks

11.2.2.1 For each extraction fluid used in a batch, prepare a method blank using the extraction fluid and 100g of clean Teflon boiling chips. Document the method blank in the worksheet in TALS.

11.2.3 Tumbling the TCLP or SPLP Sample

11.2.3.1 Close the extractor bottle **tightly** (Teflon tape can be used to ensure a tight seal).

11.2.3.2 Secure container in the rotary agitation device to keep movement of container to a minimum, and rotate at 30 ± 2 rpm for 18 ± 2 hours.

11.2.3.3 Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2°C during the extraction period. The min/max thermometer shall be set at the start of every extraction. The initial temperature and final temperature during tumbling is recorded in the TALS worksheet for both the corrected and the uncorrected reading. In addition the minimum and maximum recorded temperature for the extraction shall be recorded in the TALS worksheet for both the corrected and the uncorrected reading.

11.2.3.4 If the temperature range during an extraction falls outside the method-specified range of 23 ± 2°C, initiate a non-conformance report as described in TA-QA-0610.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

11.2.3.5 Following the 18 ± 2 hour extraction, remove containers and allow samples to settle. This will aid in filtering. Go to Section 11.2.4.

11.2.4 Filtering TCLP or SPLP extracts.

11.2.4.1 If the extract is to be analyzed for metals, use the acid-washed filter specified in section 7.3.4 or 7.3.3. Due to random Barium contamination another pre-rinse with 2% Nitric acid must be preformed. Organic extracts are filtered using the positive pressure procedure. Gravity filtering is an option if necessary due to equipment issues but is more time consuming.

11.2.5 Gravity Filtration for metals:

11.2.4.2 Place a glass funnel over a 200ml plastic beaker. Fold a TCLP filter and place in the funnel. Pre-rinse with the 2% Nitric acid solution making sure to saturate the entire filter paper with approximately 20mls of solution. Discard of the rinse waste in the neutralization tank.

11.2.4.3 Make sure to check and document the final pH of the extract from the unfiltered leachate.

11.2.4.4 Fill the funnel with leachate being careful to not fill over the top of the filter paper. Continue until the desired volume for digestion is reached.

Filtration by positive pressure

11.2.4.5 Filter sample by pouring it into filter apparatus containing a .6 to .8 micron glass fiber filter and sealing. Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

11.2.5.1 Collect the filtrate in 500 or 1L amber bottles.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

Note: For final filtration of the TCLP or SPLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

11.2.6 Handling the TCLP or SPLP Extracts.

11.2.6.1 If the sample contained no initial liquid phase, the filtered liquid material is defined as the TCLP extract.

11.2.6.2 If the sample is multiphasic:

11.2.6.2.1 Check the miscibility of the solid phase filtrate with the liquid phase filtrate(s). Pour ~ 5 mL of one of the filtrates into a small glass container (example: a 12mL vial). With a plastic pipette drop a few mLs of the other filtrate into the container. Observe them combine. If they are compatible, (miscible), multiple phases will not result on combination.

11.2.6.2.2 If they are miscible, then combine the phases into one TCLP or SPLP extract.

11.2.6.2.3 If they are not miscible, then do not combine them. Determine the volume of the individual phases (to ± 0.5%) using a graduated cylinder. Conduct the appropriate analyses, and combine the results mathematically as described below:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

11.2.6.3 If the sample contained <0.5% solids, then its own filtrate is defined as the TCLP or SPLP extract.

11.2.6.4 Following collection of the TCLP or SPLP extract, determine the pH of the extract using a calibrated pH meter. Record the pH of the extracts to the nearest tenth of a pH unit.

11.2.6.5 Metals aliquots must be either digested immediately or acidified with nitric acid to pH<2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract shall not be acidified and the extract shall be analyzed as soon as possible.

11.2.6.5.1 If the leachate is a metals matrix spike or matrix spike duplicate, the metals matrix spike solution must be added to the leachate prior to nitric acid preservation.

11.2.6.5.2 Upon completion of the preservation step, the trace metals chemist will document the preservation of the extracts in the Metals Digestion Log.

11.2.6.6 All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP or SPLP extract shall be prepared and analyzed according to appropriate analytical methods.

11.3 ZHE Volatiles Extraction Procedure

Note: Document sample information in the worksheet in TALS.

11.3.1 Use the ZHE device to obtain TCLP or SPLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to

evaluate the mobility of non-volatile analytes, e.g. metals, pesticides, herbicides, etc.

11.3.2 The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

11.3.3 Charge the ZHE with sample only once and do not open the device until the final extract has been collected.

11.3.4 Do not allow the waste, 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 (4°C) to minimize loss of volatiles.

11.3.5 All ZHE metal parts are numbered to track possible contamination. Only use the numbered parts that agree with the vessel number.

11.3.6 Perform a 1 hour pressure check on each ZHE vessel before every use. Set it up empty as outlined below and pressurize to 50 psi. Document the test in the Pressure Check Log. (Appendix A).

11.3.7 Preparation of the extractor vessel

11.3.7.1 Pre-weigh the filtrate collection container.

11.3.7.2 Apply vacuum grease to the piston O-rings and place the ZHE piston within the ZHE body, engraved number facing down. 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 (bottom 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 (top flange) aside.

11.3.8 Calculate the weight of waste to charge ZHE vessel.

11.3.8.1 If the waste is 100% solid, weigh out a subsample (25 g. maximum) of the waste and immediately transfer it to the ZHE vessel and attach the ZHE top. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. Record the weight in TALS to the nearest tenth of a gram.

11.3.8.2 If the waste contains < 0.5% dry solids, quickly pour 100-500 mLs of the sample into the ZHE vessel. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log and in the TALS worksheet.

11.3.8.3 For wastes containing between 0.5% and 5% solids, weigh out a 500g. subsample and record the weight. Since the ZHE vessel cannot be opened during the extraction, multiple vessels may be used to extract the

sample and the extracts combined for analysis. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log.

11.3.8.4 For wastes containing ≥ 5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE (g)} = \frac{25}{\text{percent solids}} * 100$$

11.3.8.5 If particle size reduction was required as determined in section 11.1.2, prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size specified in section 11.1.2. Waste and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat. Also, exposure of the waste to the atmosphere should be kept to a minimum. Don't sieve.

Note: Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Don't allow waste slurries to stand and don't centrifuge. 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 (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 11.3.8 to determine the weight of the waste sample that will be filtered.

11.3.9 Headspace Elimination and Liquid/Solid Separation

11.3.9.1 Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood.

11.3.9.2 If the waste is liquid, at the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. Proceed to section 11.3.10.

11.3.9.3 If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to section 11.3.10.

11.3.9.4 Attach the ZHE fluid pump sample tube to the liquid inlet/outlet valve with the other end of the line at the bottom of a 40ml voa vial and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into a tared VOA vial or other appropriate filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period or the syringe is full, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the sample tube. Weigh the VOA vial or other appropriate filtrate collection container to determine the weight of the waste filtered.

11.3.9.5 The filtrate may now be either analyzed immediately or stored at 4 EC under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids} \times \text{weight of waste filtered (Section 11.3.9.4)}}{100}$$

Please Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging. An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

11.3.9.6 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

Please Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP or SPLP extraction as a solid. If the original waste contained <0.5% dry solids, this filtrate is defined as the TCLP or SPLP extract and is analyzed directly.

11.3.10 Charging the ZHE Device with TCLP or SPLP Extraction Fluid.

11.3.10.1 The equation to determine the amount of extraction fluid to add to the extractor vessel is:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

For example: 25 g of the sample was weighed out and the percent solids were calculated at 25%.

$$\text{Weight of extraction fluid (g)} = \frac{20 \times 25\% \text{ solids} \times 25 \text{ g total weight of waste filtered}}{100\%}$$

The weight of extraction fluid to add to the extraction vessel in this case is 125 g.

11.3.10.2 TCLP Fluid #1 or SPLP Fluid #3 is used exclusively for the extraction of volatiles using the ZHE. Add the appropriate amount of appropriate fluid to the solid material within the ZHE according to the following steps:

11.3.10.2.1 Pre-flush the extraction fluid lines connected to the ZHE pump with fresh extraction fluid to eliminate any air pockets from the line. Fill the graduated cylinder with the calculated weight of extraction fluid you will add. With the ZHE in the vertical position, attach the extraction fluid line from the pump directly into the liquid inlet/outlet valve.

11.3.10.2.2 Open the top flange and pump all the fluid to the extraction vessel.

11.3.10.2.3 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition ZHE in the vertical position with the liquid inlet/outlet valve on top.

10.3.10.2.4 Pressurize the ZHE to 40+PSI.

11.3.11 Tumbling the ZHE Samples

11.3.11.1 Method Blank

11.3.11.1.1 One Method Blank must be run through each extraction vessel every 20 times it is used. Vessel usage is tracked in the TCLP Vessel Use Log (see Appendix C). Using this log, select an appropriate vessel for the method blank. Document the method blank in the worksheet in TALS.

11.3.11.2 Secure container in the rotary agitation device to keep movement of container to a minimum, and rotate at 30 $\pm$ 2 rpm for 18 $\pm$ 2 hours.

11.3.11.3 Ambient temperature (i.e., temperature of room in which extraction takes place) should be maintained at 23 $\pm$ 2°C during the extraction period. The initial temperature and final temperature during tumbling is recorded in the TALS worksheet for both the corrected and the uncorrected reading. The range paper with the entire nights reading should be dated and placed in the three ring binder for future reference. If the temperature range during an extraction falls outside the method-specified range of 23 $\pm$ 2°C, initiate a non-conformance report as described in B-SOP-QAG-010.

11.3.11.4 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by reading the pressure meter on the ZHE body. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Repair and retest the device or remove it from service. Initiate an NCM, then using a fresh portion of the waste, re-prepare the sample in a properly functioning device.

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. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container.

11.3.12 Filtering the ZHE Extracts

11.3.12.1 Attach the sample tube line to the liquid inlet/outlet valve with the other end of the tube placed at the bottom of a 40ml voa vial and open the valve. Begin applying gentle pressure of 1-10 psi to force the TCLP liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period or the voa vial is full, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the sample tube line. Express any additional liquid phase into a TCLP waste container once the VOA vial is full.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging. An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

11.3.13 Handling the ZHE Extracts

11.3.13.1 If the sample contained no initial liquid phase, the filtered liquid material is defined as the TCLP or SPLP extract.

11.3.13.2 If the sample is multiphasic:

11.3.13.2.1 Check the miscibility of the solid phase filtrate with the liquid phase filtrate(s) by combining via transfer pipette small portions of each component in a small glass container. If they are compatible, (miscible), multiple phases will not result on combination.

11.3.13.2.2 If they are miscible, then combine the phases into one voa vial as one ZHE extract.

11.3.13.2.3 If they are not miscible, then do not combine them. Determine the volume of the individual phases (to ± 0.5%) while contained in their respective syringes. Conduct the appropriate analyses, and combine the results mathematically as described below:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

11.3.13.3 If the sample contained <0.5% solids, then its own filtrate is defined as the ZHE extract.

11.3.13.4 The liquid phase may now be either analyzed or stored at 4°C under minimal headspace conditions until time of analysis.

12.0 Calculations / Data Reduction

12.1 Percent Solids Determination

$$\%\ \text{Solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

12.2 Percent Dry Solids Determination

$$\%\ \text{Dry Solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste}} \times 100$$

12.3 The equation to determine the amount of extraction fluid to add to the extractor vessel is:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

12.4 Final Analyte Concentration from multiple phases

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

12.5 ZHE waste amount for solids

$$\text{Weight of waste to charge ZHE (g)} = \frac{25}{\text{percent solids}} \times 100$$

13.0 Method Performance

13.1 A Method Detection Limit (MDL) study must be performed annually according to the current SOP for MDL completion (TA-QA-0602).

13.2 An IDC must be completed according to the procedures specified in TA-QA-0617.

14.0 Pollution Control Waste

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., processing one set of MDLs on all applicable instruments, examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Employees will abide by this method and the policies in the Corporate Safety Manual for "Waste Management and Pollution Prevention". Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. The lab's waste disposal procedures are incorporated by reference to TA-EHS-0036.

15.1 Waste Streams Produced by the Method.

The following waste streams are produced when this method is carried out:

15.1.1 Extraction Fluid. The remaining extraction fluid is decanted into the acid waste drum located in the waste warehouse. This waste stream is sent out for waste water treatment. The remaining solid wastes is removed from the container and placed in the toxic solid debris waste stream. This waste stream is sent out for incineration. The empty container is then placed in the dumpster and sent to the sanitary landfill.

16.0 References

16.1 TestAmerica Seattle Quality Assurance Manual (QAM), current version.

16.2 TestAmerica Environmental Health and Safety Manual, current version.

16.3 US EPA Method 1311, "Toxicity Characteristic Leaching Procedure (TCLP)", SW-846, Rev. 0, July 1992.

16.4 US EPA Method 1312, "Synthetic Precipitation Leaching Procedure (TCLP)", SW-846, Rev. 0, September 1994

16.5 TCLP Flowchart, Genium Publishing Corp., 1997.

17.0 Attachments

17.1 Appendix A: ZHE Pressure Check Log

17.2 Appendix B: TCLP Tumbler Log

17.3 Appendix C: ZHE Vessel Use Log

17.4 Appendix D: pH Meter Log

17.5 Appendix E: TCLP Flowchart

17.6 Appendix F: 1311 TCLP/ZHE Summary

18.0 Discrepancies to the Method

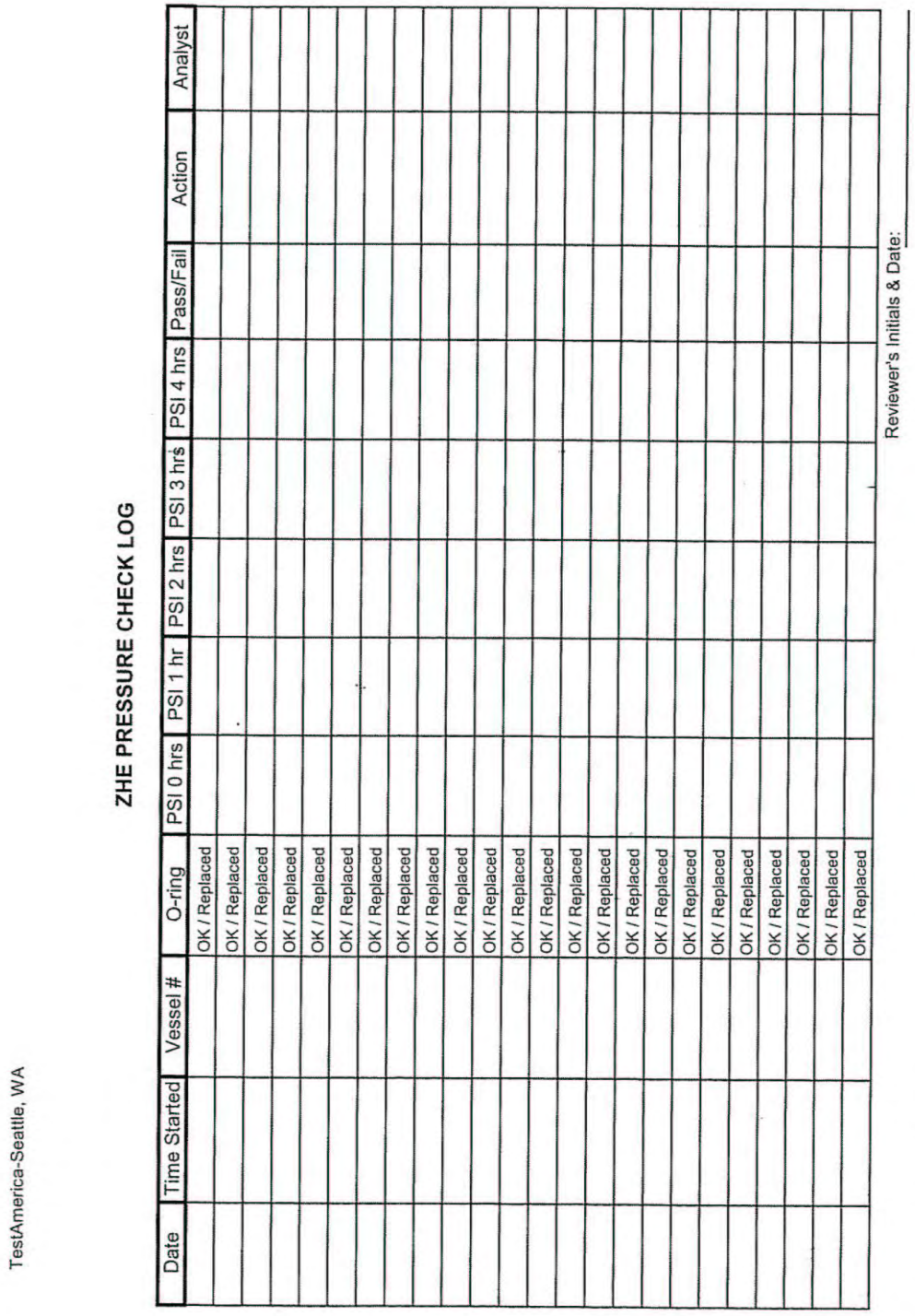
18.1 2% Nitric Acid solution is used to acid washing filters for metals mobility instead of 1 N Nitric Acid in the method due to observed Barium contamination when using the less concentrated 1 N Nitric Acid.

19.0 Revision History

- Revision 16, dated 4/18/2017
  - Added wording "as needed" to section 6.4
  - Updated pH meter low calibration point to 2.0, section 7.4.1.2 and 8.1
  - Clarified reagents in section 8.0.
  - Updated section 18.0.
- Revision 15, dated 3/1/2016
  - Added option to digest filtrate immediately, Section 11.2.6.4
  - Incorporated ROMD 00061, sections 11.1.3.1.1
  - Re-worded section 11.3.9.4 and added section 11.3.9.5 to make the requirements for ZHE samples with expressible liquid clearer.
- Revision 14, dated 11/28/2014
  - Added comments to tag non-working ZHE bodies as "out of service", Section 6.2.4
  - Added option for 68 oz containers for both plastic and glass TCLP bodies, Sections 7.1.2 and 7.13

- Specified need for 2 MB's if both extraction fluids are used in a batch, Section 10.1.1
- Added option for 68 oz containers for both plastic and glass TCLP bodies, Table 1

- Revision 12, dated 5/7/2012
  - Updated filter brands, Section 7.3
  - Removed reference to using EE tube filtration process, throughout
  - Added rinse solution for filters and reagent documentation to be done in TALS, section 8.0
  - Made reference to LCS/LCSD batching and added requirement for MSD, section 10.0
  - Added gravity filtration process and pre-rinse step for filter paper for metals, section 11.0
  - Added Appendix F
- Revision 11, dated 4/25/2011
  - Changed documentation requirements from TCLP logbook to TALS worksheet , throughout
  - Incorporated ROMD 00021, sections 11.2.3 and 11.3.11.3
  - Deleted Attachments C (TCLP Log) and D (TCLP Vessel Use Log) because of documentation and vessel change
- Revision 10, dated 3/10/2010
  - Added documentation of standards/reagents and standard/reagent preparation, section 8.0
  - Added removal of expired standards, section 8.10
  - Addressed the need to evaluate extra QC when present, section 10.4
  - Added daily balance check, section 11.1



Appendix A: Example of a ZHE Pressure Check Log

TestAmerica-Seattle, WA

ZHE PRESSURE CHECK LOG

Date	Time Started	Vessel #	O-ring	PSI 0 hrs	PSI 1 hr	PSI 2 hrs	PSI 3 hrs	PSI 4 hrs	Pass/Fail	Action	Analyst
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								

Reviewer's Initials & Date: ______________

B-NBK-EXT-132

Page 120 of 120

Appendix B: Example of a TCLP Tumbler Log

Tumbler Check should be performed on a monthly basis, while fully loaded

Date	Tumbler #1		Analyst
	RPM	Action	

Date	Tumbler #2		Analyst
	RPM	Action	

Date	Tumbler #3		Analyst
	RPM	Action	

Appendix C: Example of ZHE Vessel Use Log

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Date:																				
Batch:																				
ZHE#	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**	**S/B**
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				

S=Sample B=Blank

Appendix D: Example of pH Meter Calibration Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

pH 3 - Calibration Logbook

Analyst________________ Date___________________

Calibration

pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Analyst________________ Date___________________

Calibration

pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Analyst________________ Date___________________

Calibration

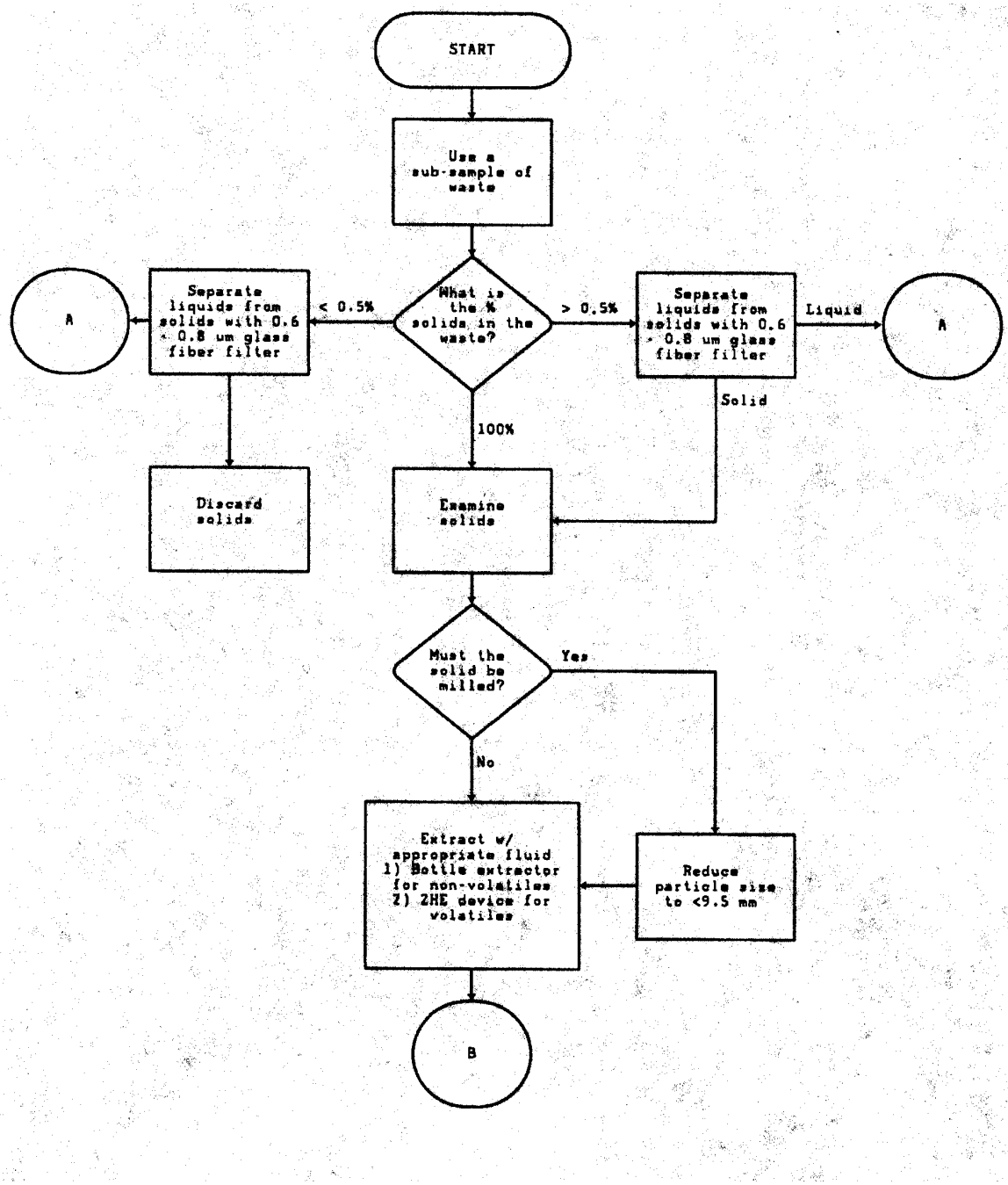
pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Analyst________________ Date___________________

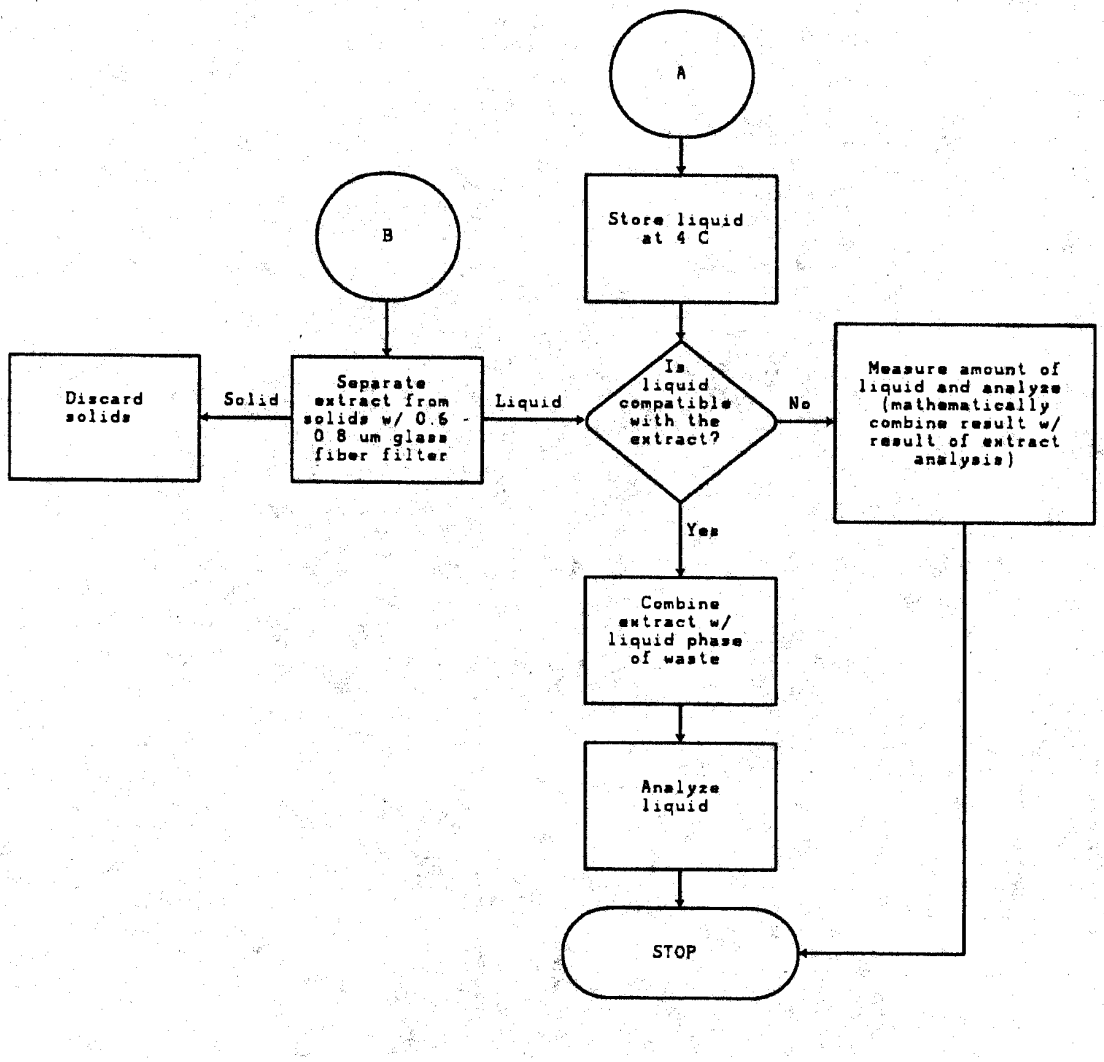
Calibration

pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Appendix E: TCLP Flowchart



Appendix E: TCLP Flowchart (continued)



Appendix F: 1311 TCLP/ZHE Summary

Quarterly checks:	Pipettor calibration
Monthly checks:	RPM check for tumbler while full, 30 ± 2 RPM required
	4 hour pressure check for all working ZHE bodies
Daily Checks:	Daily temperature control unit maintenance: Drain and dump water waste
	pH probe calibration and fluid pH verification, 4.88 to 4.98

1311 Metals, 8270, 8081, 8151

If the sample is not obviously 100% water or 100% solids:

Percent solids determination:
First weight the sample container and entire contents of the jar.
This weight is the **Total Initial Volume.**
Set up the pressure filter device. Transfer the entire contents of the sample jar into the filtration device.
Weigh the empty jar, this is the **Container weight**.
Total Initial Volume – Container weight = Total sample weight
Weigh an **Empty container**, glass or plastic.
Pressurize the filtration device and capture the filtrate in this container.
Weigh the **Full container** when complete.
Full container – Empty container = Total aqueous weight.
Total soil weight = Total sample weight – Total aqueous weight

% solids = (Total soil weight / Total sample weight) x 100
Check SOP for complete details on re-combining portions

Water samples are filtered only in plastic 200ml beakers for metals and amber bottles for Organics

Soil samples are first pH tested (already determined 100% solid)
Determine if particle size reduction is necessary, material should be broken, cut, torn apart into 1cm³ portions before continuing.

5grams – add DI water up to 101.5 grams
Stir and take pH
If pH is ≤ 5 you're done skip to fluid addition of fluid #1
If pH is >5 then put on the hot plate
Add 3.5 mls of 1N HCL
Heat to 50 degrees +/- 5 degrees keep in range for 10 minutes
Let cool approx 10 minutes
Take the pH
If pH ≤ 5 use TCLP fluid #1
If pH > 5 use TCLP fluid #2
100 grams of soil/solid to 2000mls of fluid
Samples are ready to be tumbled for 18 ± 2 hours

Quality Control:
MB per batch of 20 samples

MB for each fluid type per batch
Corrected Temperature out of range of 21-25 degrees – **NCM**
100grams not available – **NCM** and maintain a 20:1 fluid:sample ratio.
Any required checks not completed – **NCM** and immediately complete check

Appendix F: 1311 TCLP/ZHE Summary (continued)

Filtration Process

Metals –
For MB and sample to be used as Matrix QC filter full 200mls
For the rest of the samples at least 100mls
Gravity filter using a glass funnel and TCLP filter. Pre-rinse the filter using ~ 20mls of the 2% Nitric acid solution. Discard waste before filtering the sample.
Take the final leachate pH on a portion of **un**-filtered leachate
Deliver to metals

8270, 8081, and 8151
Use the positive pressure filtration device with a TCLP filter in place and filter ~ 1L into an amber bottle.
Store in walk-in on designated shelf

1311 ZHE for Volatile Analysis

If the sample is not obviously 100% water or 100% solids:

The determination is the same as for non-volatile analysis except the filtration device is the ZHE body itself.

Water samples are filtered only through a ZHE body complete with TCLP Filter.

Soil samples determined to be 100% solid
Determine if particle size reduction is necessary, material should be broken, cut, torn apart into 1cm³ portions before continuing.

25 grams of soil is placed in the ZHE body
Pressurize the ZHE and remove any headspace by opening the top valve allowing the piston to move up
Add 500mls of TCLP fluid #1 to a class A graduated cylinder
Attach the tubing from the pump to the top of the ZHE body and place the other end of the tubing in the graduated cylinder
Pump the fluid into the ZHE body
Re-pressurize to over 40psi if necessary
Samples are ready to be tumbled for 18 ± 2 hours

Quality Control:
Same as Non-volatile portion
If pressure is lost overnight – **NCM** and possible re-preparation required

Filtration Process

Attach the filtration tubing to the top of the ZHE body
Place the other end of the tube at the bottom of a 40ml voa vial
Fill the voa vial to the top and cap with no head space
Deliver to volatile's analyst or store in voas refrigerator

Appendix F: 1311 TCLP/ZHE Summary (continued)

Fluid Preparation:

TCLP Fluid #1 Concentrate:

In a 20L carboy add 10 L of DI water
Weigh 1040 grams of Sodium Hydroxide pellets
Slowly add the pellets to the carboy adding only approximately 1/4th of the total volume at one time
Cap the carboy and roll on the counter to mix
Let cool and add another 1/4th volume of the pellets and repeat the mixing and cooling step two more times
When all the sodium hydroxide is dissolved and cooled to near room temperature you can proceed
Slowly add 2.3 liters of glacial acetic acid
Fill to the 20L mark on the carboy and mix well
Let cool-mix again and read the pH
Should fall in the range of 4.88 to 4.98

TCLP Fluid #1:

Dilute 1 Liter of concentrate to 20L with DI water in a carboy
Mix well and verify pH between 4.88 and 4.98

TCLP Fluid #2:

Add 22.8 mls of glacial acetic acid to 4 Liters of DI water
Mix and verify pH between 2.83 and 2.93

For more details including preparation for SPLP the SOP must be referenced. This summary is not intended to replace the SOP and any details not referenced with in this summary should be sought out in the SOP.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS [Method 8270D]

Approvals

Signatures on File	
Joan Protasio Date	Manjit Nijjar Date
Semivolatile Organic Department Manager	Health & Safety Manager / Coordinator
Terri Torres Date	Dennis Bean Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270D, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 Direct injection of a sample may be used in limited applications.

1.1.3 Refer to Table 1 for the list of compounds applicable for this method. This method may be amenable to additional compounds. If non-standard analytes are required, they must be validated by the procedures described in section 12 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3- and 4-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.

1.3 The reporting limit (RL) of this method for determining an individual compound is approximately *10 ug/kg to 4,000 ug/kg* for soil/sediment samples and *0.02 µg/L to 15 µg/L* for water samples. Some compounds have higher reporting limits. The current reporting limits are all updated in TALS. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor *or Separatory Funnel.*

2.2 Solid samples are extracted with methylene chloride / acetone using sonication *or microwave extraction.* The extract is dried, concentrated, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

TA-OP-0302 SONICATION EXTRACTION PROCEDURE, SW846 3550B

TA-OP-0323 CONTINUOUS LIQUID-LIQUID EXTRACTION, SW846 3520C

2.5 *Additional extraction procedures are detailed in the following SOPs. They may be subject to having complete MDL studies so check with QA before using.*

TA-OP-0301 Separatory Funnel Extraction (3510C)

TA-OP-0367 Microwave Extraction (3546)

2.6 Qualitative identification of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.0 Definitions

3.1 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica Seattle SOP TA-QA-0620 Quality Control Program for further details of the batch definition.

3.2 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.3 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.4 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.5 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.6 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples.
- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section 9.0). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum. Cut resistant gloves must be worn when using sharp tools or when washing glassware.

5.1 Specific Safety Concerns

5.1.1 Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: LATEX AND VINYL GLOVES PROVIDE NO PROTECTION AGAINST THE ORGANIC SOLVENTS USED IN THIS METHOD. NITRILE OR SIMILAR GLOVES MUST BE USED.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass

spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power before performing any maintenance.

5.1.5 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Significant or Serious Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source
- Mass Spectrometer: Capable of scanning from 35 to 500 u (previously “amu”) every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 3 when 25 ng of the GC/MS tuning standard is injected through the GC
- Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent
- Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.
- GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used
- Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have 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 the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the NIST Mass Spectral Library is recommended. Agilent (Hewlett Packard) ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent’s ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.
- Data processing: Chrom version 1.2 or higher.
- LIMS system: TALS version 1.0 or higher

6.2 Supplies

- Column: 30 m x 0.25 mm I.D., 0.25-µm film thickness fused-silica capillary column *(Phenomenex ZB-SemiVolatiles, Restek Rxi-5SilMS, or equivalent).*

  Note: Other columns may be used. This was the column in place at the time the SOP was prepared. The serial number of the column used is documented in the instrument maintenance logbook.
- Gas-tight syringes (Hamilton 1700 Series, or 1000 Series or equivalent).
- 10 ml scintillation vials with polypropylene closures or 10, 20, 40 or 60 ml VOA vials with Teflon-lined silicone septa enclosures (or equivalent).
- Analytical balance, capable of reading to 0.0001g. Analysts must verify calibration has been preformed on the balance before using it. The calibration must bracket the weights to be determined.
- Class A volumetric flasks; 10 mL, 25 mL, 50 mL, 100 mL, 250 mL.
- Carrier gas: Ultra high-purity helium

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2.1 Initial calibration stock standards 8270

7.2.1.1 *1000 ug/ml 8270 List 1 / Std#1 MegaMix – Restek 571995*

2000 ug/ml 8270 List 1 / Std#9 - Restek 569730

2000 ug/ml 8270 List 1 / Std#10 - Restsk 569731

2000 ug/ml 8270 List 1 / Std#11 - Restek 569732

5000 ug/ml Surrogate stock – Phenova AL0-130068

7.2.2 Intermediate calibration standard 8270

7.2.2.1 Dilute 1.0-mL of stock solutions listed in section 7.2.1.1. For the surrogate standard, dilute 200 uL. Mix to a final volume of 10-mL to make a 100-500 µg/mL intermediate stock solution.

7.2.3 Working calibration standards 8270

7.2.3.1 Dilute the intermediate calibration stock solution (section 7.2.3) as follows to make working calibration standards:

Calibration Level	Volume of Intermediate Stock (µL)	Final Volume	Concentration (µg/L)
1	5	50	10
2	10	50	20
3	25	50	50
4	50	50	100
5	100	50	200
6	250	50	500
7	500	50	1000
8	1000	50	2000
9	2500	50	5000
10	5000	50	10000

7.2.4 ICV Standard 8270

7.2.4.1 *8270 List 1 / Std#1 MegaMix – Restek 571995.SEC*

8270 List 1 / Std#9 - Restek 569730.SEC

8270 List 1 / Std#10 - Restsk 569731.SEC

8270 List 1 / Std#11 - Restek 569732.SEC

7.2.4.2 1.0 ug/mL ICV Working Solution is prepared by diluting 100 µL of each standard in section 7.2.4.1 to a final volume of 100-mL with methylene chloride.

7.3 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.3.1 2000 ug/ml 8270 Internal Standard Restek #567684. A 100 ug/ml 8270 Internal Standard is prepared by diluting 5.0 ml of the 2000 ug/ml Restek Standard to 100 ml with methylene chloride.

7.3.2 Internal standards are added to all standards and extracts to result in a final concentration of 1000 µg/L for full scan and 100 µg/L for SIM. For example, if the volume of an extract aliquot used was 1 mL, 10 µL of a 100 µg/mL internal standard solution would be added to the aliquot.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 25 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 25 µg/mL. 2uL of this solution should be injected for an on column concentration of 50ng.

7.5 Laboratory Control Spiking Solution, Matrix Spike Solution, and surrogate spike solutions: Prepare as indicated in the extraction SOPs.

7.6 The standards listed in sections 7.1 to **Error! Reference source not found.** must be refrigerated at 0-6°C. Stock standards expire 1 year after preparation.

7.7 ICAL reagents should be replaced after one month if the vials have been opened frequently (more than 5 times in a month).

7.8 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in pre-cleaned, amber glass bottles fitted with a Teflon-lined cap. To achieve routine reporting limits, a full one-liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

8.2 Alternatively, water samples can be collected in a pre-cleaned, amber 250mL bottle with a Teflon-lined cap. *Currently, LVI volumes are only used for 8270 SIM PAH analysis.*

8.3 Soil samples are collected in 8-ounce, pre-cleaned, wide-mouth jars with a Teflon-lined lid.

8.4 Samples and extracts are stored at 0-6°C.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	250mL for LVI or 1 Liter for non LVI	Cool 0-6°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	Glass	30 grams	Cool 0-6°C	14 Days	40 Days from extraction	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section and in Table 6. The process of establishing control limits, and the use of control charts are described more completely in TA-QA-0620, Quality Control Program. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in SOP TA-QA-0620, Quality Control Program.

9.1.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via instructions in the LIMS.

9.1.3 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed together using the same reagents and standards. Each quality control batch must contain a method blank (MB), a laboratory control sample (LCS), matrix spike (MS), and/or matrix spike duplicate (MSD) or duplicate (DUP) pair. For more details see SOP TA-QA-0620.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher.

NOTE: Some programs (e.g., DOD and BP) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. For DOD and BP, an LCSD must be analyzed if there is not sufficient volume for a MS/MSD. The LCSD must pass the same control criteria as the LCS. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See QC SOP TA-QA-0620 for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the LCS recovery is high and there are non-detect samples. An NCM is initiated. The non-detect samples are flagged and reported.

  NOTE: DOD programs do not allow reporting data from high LCS's with sample non-detects. If data is to be reported, it must be authorized by the client via a variance on a site by site basis.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the

project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS. An MS/MSD pair is prepared and analyzed with every batch of samples when sufficient sample volume is available.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See QC SPP TA-QA-0620 for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.6 Surrogates

9.6.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds are spiked at 100 µg/mL. The compounds routinely included in the surrogate spiking solution, along with recommended standard

concentrations, are listed in Table 4.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: For particular sublists, such as PAH only, acid surrogates may fail with no corrective action required. However, the failure must be documented in an NCM if the surrogates are reported. Otherwise, if any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Re-extract and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem.

Note: For BP LaMP samples, if the surrogate %R fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the BP representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

NOTE: The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/ reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.6.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.6.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.6.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.7 Instrument QC

9.8 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP TA-QA-0610.

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

TA-OP-0302 SONICATION EXTRACTION PROCEDURE, SW846 3550B

TA-OP-0323 CONTINUOUS LIQUID-LIQUID EXTRACTION, SW846 3520C

Additional extraction procedures are detailed in the following SOPs.

TA-OP-0301 Separatory Funnel Extraction (3510C)

TA-OP-0367 Microwave Extraction (3546)

TA-OP-0314 Waste Dilution (3580A)

10.2 Instrument Operating Conditions

10.2.1 Typical instrument operating conditions are listed in Table 2. Actual instrument operating conditions are posted in each maintenance logbook.

10.2.2 The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s).

10.2.3 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 8270 SIM PAH

10.3.1 SIM (selective ion monitoring) is an alternative to analyzing samples under full scan mode. SIM selects specific target ions for analysis. SIM can be up to ten times more sensitive. In order to achieve maximum sensitivity the selected ions should be broken up in to several groups. *Each of the target analytes should have 1 ion used for quantitation and 2 qualifier ions. The suggested SIM groupings and ion are in Table 7.* Other parameters can be used as long as sufficient sensitivity is achieved.

10.3.1.1 SIM can also be extended to other 8270 target analytes not listed in Table 7.

10.3.1.2 *The internal standards and surrogates do not need to have 2 qualifier ions.*

10.4 Instrument Tuning

10.4.1 A MS tuning compound (DFTPP) is analyzed every twelve hours during instrument operation, prior to analysis of standards, samples, or QC samples. Method tuning criteria must be met before sample analysis can proceed.

10.4.2 Tuning Procedure (Ion Trap): 2.0 ul of a 2.5 ng/uL solution of decafluorotriphenylphosphine (DFTPP) must be analyzed in a scanning mode of 40 - 450 m/z. The tuning solution must also contain 4,4'-DDT, Pentachlorophenol, and Benzidine at the same concentration.

10.4.3 Tuning Procedure (Quadrapole): 2.0 ul of a 25 ng/uL solution of decafluorotriphenylphosphine (DFTPP) must be analyzed in a scanning mode of 40 - 450 m/z. The tuning solution must also contain 4,4'-DDT, Pentachlorophenol, and Benzidine at the same concentration.

10.4.4 Inject the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed. DFTPP Tuning Criteria (per EPA method 525.1):

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

10.4.5 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Column performance and Injector Inertness Acceptance Criteria:

- Benzidine tailing factor of $\leq$ 2.0
- Pentachlorophenol tailing factor of $\leq$ 2.0
- Degradation of 4,4'-DDT to 4,4'-DDE and 4,4'-DDD < 20%.

10.5 Initial Calibration

10.5.1 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Section 7.6. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation. 10 uL of internal standard solution is added for every 1 mL of extract to all calibration standards, QC samples, and samples prior to

analysis. The autosampler injects up to 5 uL of standard and extract volumes into the instrument for analysis.

10.5.2 Compounds are assigned to the IS with the closest retention time.

10.5.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest when average response factors or linear regression curve fits are used. Six standards must be used for a quadratic least-squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response.

10.5.4 Rejection of Calibration Points

10.5.4.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.5.4.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then points may be rejected, but only if all of the following conditions are met:

- The rejected point(s) are the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
- The lowest remaining calibration point is still at or below the project reporting limit; and
- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.5.5 Add the internal standard mixture to result in a 1,000-µg/L final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 5 µL of the 100 µg/L internal standard).

10.5.6 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and Corporate SOP CA-Q-S-005. No sample analysis may be performed unless these criteria are met.

Resolution check.

Isomeric pairs need have greater than >50% resolution. Benzo(b)fluoranthene and benzo(k)fluoranthene are usually the most likely failure. This isomeric pair must have >50% resolution. This needs to be checked on the mid point ICAL and the CCV. If the

combined peak “benzofluoranthenes “ is being requested, the 50% resolution check is not applicable, but all other isomers that are in the list must also be >50% resolution.

10.5.7 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with RSD > 20% (>15% for DOD)for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

10.5.8 If the RSD for a compound in the initial calibration is > 20%(>15% for DOD), then calibration using a curve fit, must be used. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.5.9 If a linear regression equation is used, the correlation coefficient (r) must be greater than 0.990 for commercial projects and greater than 0.995 for DoD projects, and r squared (r2) greater than 0.990.

10.5.10 Use of second-order equations (quadratic) may be used on rare occasions and must consist of a minimum of six data points. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination (r2) must be greater than 0.990.

10.5.11 Weighting of Calibration Data Points

10.5.12 In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration2 weighting (often called 1/X2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5.13 In addition to meeting %RSD requirements, each analyte must meet a minimum RF requirement. The minimum RF requirements are defined in table 8.

10.5.14 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

10.5.15 An initial calibration verification containing all components from a second source (an alternate vendor or a unique lot from the same vendor) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 80-120% of all target analytes for DoD (e.g., Navy and USACE) and BP LaMP projects; 70-130% for non-DoD projects (e.g., 8270D HSL components); and 50-150% for poor performers (see Table 5).

10.5.16 If the percent difference for the second-source verification falls outside acceptance criteria, then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result fails, then re-prepare the verification standard, and/or re-prepare and rerun the ICAL.

10.5.17 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.6.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.5.18 If a calibration is in use for greater than 3 months a check at the low and high ends of the calibration will be performed once per month of continued use and the %D and evidence of saturation for the high end check will be used to evaluate the continued effectiveness of the calibration.

10.6 Continuing Calibration Verification (CCV)

10.6.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 3.

10.6.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the CCV.

10.6.3 The following criteria must be met for the CCV to be acceptable:

- For DOD samples, the percent difference or drift (%D) must be within ± 20% for all reported analytes. Any samples associated with a continuing calibration verification standard where the response for an analyte in the verification standard is above the acceptance limit and the analyte is not detected in any of the samples analyzed in the 12-hour window, do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected if it were present (for DOD samples this requires client pre-approval). If a compound in the CCV fails low, the analyst may elect to analyze a RL (CCVL)standard immediately after the CCV. If the compounds of concern are detected in the RL standard, it demonstrates that they would be detected in the samples, if present. This allows for the reporting of non detect sample results. Any compounds using a linear calibration fit in the initial calibration must undergo a low level readback on the CCVL. The readback concentration must be within 30% of the true value unless the analyte has been identified as a poor performer in which case the readback value must be within 50% of the true value. Compounds failing the readback value must be re-analyzed. For situations where the failed compound is present in a sample, the results must be qualified or the problem must be fixed and the CCV and affected samples must be re-analyzed. Possible problems include standard mixture degradation, column contamination and active sites.

  If the subsequent calibration verification injection fails, a new initial calibration curve must be processed. (i.e., no more than two consecutive injections of the calibration verification may be processed.

- Analysis of DOD samples also requires a closing CCV to be analyzed at the end of the analytical run. Closing CCV requirements are 50-150%D for all analytes.

- For non-DOD samples, all compounds listed in Table 1 must meet 20%D except those listed as poorly performing compounds in Table 5 which must be within ± 50%D. (See Section 12 for calculations)
- For SIM PAH analysis of samples analyzed under the BP Lamp program, all target analytes must meet ± 15% D. See above for corrective actions.
- For non BP SIM samples, the percent drift must be ± 20% for all compounds.

  NOTE: Some analytes are included in both Tables 1 and 5. Those analytes that are in Table 1 will be controlled to ± 20% for projects reported under the DoD QSM and will be controlled to ± 50%D for commercial projects.
- The internal standard response of the CCV must be within 50 - 100% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

10.6.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

10.7 Sample Analysis

10.7.1 Calibrate the instrument as described in Section 10.5. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.7.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.7.3 Add internal standard to an aliquot of the extract to result in a 1000-µg/L concentration (for example, 10 µL of internal standard solution at 100 µg/mL in 1000 µL of extract). Mix thoroughly before injection into the instrument. The internal standard response must be within 50-100% of the response in the daily CCVIS.

10.7.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.7.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 11. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.7.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see Corporate SOP CA-Q-S-002) or automatically by the data system. *Chrom generates a report of the before and after chromatograms.*

10.7.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 11.1.

10.7.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 11.2.

10.8 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the midrange of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits and the matrix allows for analysis at a lesser dilution, the sample may be reanalyzed at a lesser dilution.

10.8.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample may be reanalyzed at a more concentrated dilution. **This requirement is approximate and subject to analyst judgment.** For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.8.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.9 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 0-6°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.10 Retention Time Criteria for Samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.11 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP TA-WC-0125 for determination of percent moisture.

10.12 Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.13 Maintenance Guide for GC/MS systems

10.13.1 Routine Instrument Maintenance

In addition to the schedules listed in Appendix A, the following activities constitute routine maintenance procedures and are performed as necessary.

- Clip Column;
- Install new injection port liner;
- Install new septum;
- Install new gold seal and washer, or equivalent;

10.13.2 Injector port maintenance is performed whenever the following conditions exist:

- High column bleed
- Peak broadening and/or tailing for polar analytes such as phenols
- Loss of sensitivity
- Calibration failures due to a loss of response
- Retention time drift
- Long or training solvent tail
- Overall loss of instrument response

10.13.2.1 Turn the GC oven off and let the system cool to room temperature. Remove the column nut and column from the injector body. Remove the injector nut, removing the septum and liner from the injector body. (See Illustration 6-10 in the instrument manual).

10.13.2.2 Clean the inside of the injector body with a cotton swab dipped in methanol. Follow with a wash of methanol, collecting the washings below at the column inlet port. Allow to air dry, and then replace the liner with a new or reconditioned liner that has been boiled in mineral acid, solvent rinsed, and muffled at 400°C. Replace the septum and tighten the nut just past finger tight.

10.13.2.3 Using a ceramic column cutter, remove at least 4 cm of the column end, depending on the severity of the system contamination. Place a column nut and new ferrule over the end of the column and re-cut one inch from the column end to ensure that no ferrule fragments remain in the column. Feed the column into the tapered liner until seated, then hold pressure on the column while the nut is tightened to one turn past finger tight. At this point, the GC oven is turned on and brought up to operating temperature. The system should then be leak checked.

10.13.3 Column installation is performed when the following conditions are encountered;

- Heavy column bleed that cannot be eliminated by thermal conditioning.
- Loss of early eluting peaks due to column cutting.
- Inability to chromatographically resolve method performance compound peaks (i.e. chrysene from benzo(a)anthracene).
- Distortion of peak shapes i.e.; broadening, ghost peaks, split peaks that can't be resolved by injection port maintenance or flow control.

10.13.3.1 Turn the GC oven off and let the system cool to room temperature. Remove the column nut, liner, septum, and presstight inlet connector. Dispose of old column appropriately.

10.13.3.2 Cut approximately six inches off of the end of new columns (DB5-MS 30m, 0.1u film thickness). Attach the column to the presstight inlet connector on the injector end and proceed as in 5.3.1.4 to connect to the injector.

10.13.3.3 Turn the GC on and set the injector temperature to 280°C. Allow helium to flow through the column for a couple minutes, and then turn the oven to 310°C and condition for at least an hour.

10.13.3.4 Perform a leak check on the system following the instructions contained in the operator's manual chapter on **Miscellaneous Procedures of Operation**. When the air water spectrum shows acceptable levels, proceed with the mass calibration procedure. For additional information of column replacement see the operator's manual chapter on **Selected Routine GC Maintenance** (pages 6-33 to 6-41).

10.13.4 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the ion volume or repeller, cleaning the source, replacing the multiplier, and replacing the "top board" or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

10.13.5 Autotune the MS

After major maintenance an autotune of the MS must be performed. Using an Agilent 5973 or 5975 MS, Select Autotune and run a tune to tune the MS.

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, tune, etc as may be the case).

If columns were replaced during maintenance procedures the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook.

10.14 Troubleshooting

1. If a DFTPP tune fails spectra, replace vial with fresh tuning solution and reanalyze the tune sample
    a. If it fails a second time evaluate, MS conditions
    b. Continued failures may result in re-auto tuning the instrument (10.13.5)
2. If tailing fails for either benzidine or PCP, minimum routine maintenance is required (see section 10.13.)
    a. Continued failure. Check column positioning into the source
    b. Replace column if all other options are exhausted
3. If DDT breakdown fails, minimum routine maintenance is required
    a. Continued failure. Check column positioning into the source
    b. Replace column if all other options are exhausted
4. IF CCV fails for TC target analytes, re-analyze a fresh CCV, if it fails a second time minimum routine maintenance is required. If the 2nd CCV is acceptable, the samples may be analyzed

a. A second CCV failure requires additional instrument maintenance and generating a new ICAL

10.15 Examples of Analytical Sequences:

Example 1
RB
DFTPP
STD IC 10 8270
STD IC 20 8270
STD IC 50 8270
STD IC 100 8270
STD IC 200 8270
STD IC 500 8270
STD IC 1000 8270
STD IC 2000 8270
STD IC 5000 8270
STD IC 10000 8270
ICV
QC and Samples (up to a 12 hour time limit)

Example 2
RB
DFTPP
CCV
QC and Samples (and CCVC if DOD) up to a 12 hour time limit

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST library**.** Two criteria are used to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: *Sometimes extract matrix and high targets can cause the analytes to shift outside the retention time found in the CCV. Identification can still be determined using the characteristic ions. Also, dilutions to lessen the matrix effects may be necessary to verify the identification.*

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

11.1.1 Full Scan Analysis

11.1.1.1 The sample component retention time must compare to within ± 0.06 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

11.1.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

11.1.1.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%).

11.1.1.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

11.1.2 SIM Analysis

The reference mass spectrum must be generated using the conditions of this method on the same instrument used for sample analysis. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions >30% relative intensity, if less than three such ions occur in the reference spectrum. The mass spectrum of the peak is evaluated to confirm the presence of the compound. Spectra are compared against the reference spectra of each compound by an analyst competent in the interpretation of mass spectra. The following requirements must be met:

11.1.2.1 *DFTTP tune (run in SCAN mode) runs before a 12 hour clock.*

11.1.2.2 *The quantitation and qualifier ions must be used for the identification of target compounds.* Characteristic ions for the target compounds are presented in Table 7. The monitoring ions must agree within 20% of the relative intensities of the same ions in the reference standard.

11.1.2.3 The RT of the secondary ion must elute within 2 seconds of the primary ion in the sample.

11.1.2.4 The relative RT (RRT) of the compound in the sample must be within ±0.006 RRT of the standard compound. *Matrix may affect the RT and the analyst should use their technical judgement for identification. Further dilutions may be necessary to verify identification.*

11.1.2.5 A result should be reported as non-detect if, after careful review and in the technical judgment of the mass spectral interpretation specialist, the GC/MS identification cannot be considered a qualitatively confident mass spectral identification (regardless of the concentration).

11.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches

shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

11.2.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 The relative intensities of the major ions should agree to within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

11.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

11.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

11.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylnaphthalenes
Methylphenols
Trichlorophenols
Tetrachlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b), (k), and (j)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. To begin, the isomers in a standard mix must be completely resolved (i.e., the baseline to valley height between the isomers is less than 50% of the sum of the two peak heights). Otherwise, the isomers must be identified as isomeric pairs. Next, the analyst must carefully compare the retention times between the unknown and the calibration standard.

11.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol

4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Benzidine

11.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound

SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RF_i = RF for each of the calibration levels

n = Number of RF values

11.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

11.7 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

11.7.1 Average Response Factor Calibration

If the average of all the RSDs of the response factors in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

C_{ex} = Concentration in the extract, µg/mL

R_x = Response for the analyte

R_{is} = Response for the internal standard

C_{is} = Concentration of the internal standard

$\overline{RF}$ = Average response factor

11.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

C_{ex} = Concentration in the extract, µg/mL
R_x = Response for the analyte
R_{is} = Response for the internal standard
C_{is} = Concentration of the internal standard
A = Intercept of linear calibration line
B = Slope of linear calibration line

11.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

C_{ex} = Concentration in the extract, µg/mL
R_x = Response for the analyte
R_{is} = Response for the internal standard
C_{is} = Concentration of the internal standard
A = Intercept
B = Factor for the linear term of the quadratic calibration function
C = Factor for the curvature term of the quadratic calibration function

11.8 Calculating the Concentration in the Sample

11.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration in the extract
V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)
V_o = Volume of the sample that was extracted (mL)

11.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

C_{ex} = Concentration in the extract

V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000.)

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

11.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR}	=	Spike sample result
S_R	=	Sample result
S_A	=	Spike added

11.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD	=	Relative percent difference
MS_R	=	Matrix spike result
MSD_R	=	Matrix spike duplicate result

11.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x	=	Area of the characteristic ion for the compound being measured
A_{is}	=	Area of the characteristic ion for the specific internal standard
C_x	=	Concentration of the compound being measured (µg/L)
C_{is}	=	Concentration of the specific internal standard (µg/L)

11.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (11.11) with the following exceptions:

A_x	=	Area of the total ion chromatogram for the compound being measured
A_{is}	=	Area of the total ion chromatogram for the nearest internal standard without interference
RF	=	1

11.13 Calculating Percent DDT Breakdown

$$\%\,\text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

11.14 Calculating the Peak Tailing Factor

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

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that .

AC = AB + BC, with
AB = left-hand segment
BC = right-hand segment

11.15 Upon completion of the analytical sequence:

11.15.1 Create a worklist on Chrom that reflects the machine run sequence. The Chrom worklist will serve as the instrument sequence logbook. For the Rinse Blank in the sequence, add the solvent to the sample reagent tab. This will serve as the record of the solvent lot used to dilute the samples.

11.15.2 Review chromatograms online and determine whether manual data manipulations are necessary.

11.15.3 All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

11.15.4 Manual integrations are processed *using Chrom which saves* the before and after chromatograms, the reason for the change, and attaches the analyst's electronic signature.

11.16 Compile the raw data for all the samples and QC samples in an analytical batch.

11.16.1 Perform a level 1 data review, *acknowledge any Data Review Checker (DRC) findings, and document the review on the data review checklist.*

11.16.2 Submit the review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure (see SOP TA-QA-0602). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.1.1 Instrumentation software must have each target limit set to the lowest MDL. CHROM (LOD)

12.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

12.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

12.4 Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 Acidic extracted sample and QC wastewater. After the extraction has been completed the spent water is neutralized and then collected into the organics extraction water conical reservoir. The collected wastewater is then purged with air to remove any remaining methylene chloride. The wastewater can then be discarded down the drain.

14.1.2 Methylene chloride waste. Solvent/Methylene Chloride waste. Any waste solvents are collected in beakers and then poured into a 4-liter amber bottle labeled "Hazardous Waste" located in the hood. After the extraction has been completed the MeCl2 collected in the 4 L bottles is emptied into the MeCl2 satellite waste barrel located next to the neutralization tank in lab hood #17. The funnel lid on the drum must be closed after each use At or before the satellite waste reaches 55 gallons the barrel is transferred to the waste disposal room from where it is sent out for recycling or fuel blending.

14.1.3 Vialed extract waste. Sample extracts that have been placed in vials for analysis are discarded into plastic satellite waste buckets labeled "Hazardous Waste" located underneath the bench top. Once the buckets are full the GC vials are bulked into the non-PCB GC vial waste barrel located in the waste room and sent out for incineration.

14.1.4 Extract waste. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days has passed these extracts are transported to the waste room in racks of 100 were they are bulked into a flammable loose pack waste stream and sent out for incineration.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Update IV, February 2007, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8270D.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, prepared by DoD Environmental Quality Workgroup, Final Version 5.0, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	Include method references	A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method
2	8270D	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification
3	8000B/8270D	This procedure includes the option for weighted linear regression curves using 1/concentration^2 weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on 1/standard deviation^2 weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance (standard deviation^2) is proportional to the standard concentration. EPA accepted this argument and issued a letter in July 1998, which authorizes the use of 1/concentration^2 weighting factors

17.0 Tables, Attachments, and Appendices

Table 1: Current Compounds Applicable to Method
Table 2: Suggested Instrument Conditions
Table 3: DFTPP Key Ions and Ion Abundance Criteria
Table 4: 8270D Surrogate Compounds
Table 5: Table of Poorly Performing Compounds
Table 6: Summary of QC Requirements
Table 7: Characteristic Ions – SIM

Attachment 1: Example Internal Standard Evaluation Custom Report
Attachment 2: Example Breakdown Evaluation Custom Report
Attachment 3: Example Tailing Evaluation Custom Report

APPENDIX A: Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

18.0 Revision History

- Revision 3, dated 20 April 2017
  - Removed mention of low-level analysis and updated RL ranges, section 1.3
  - Added additional extraction methods, sections 2.0 and 10.1
  - Removed PIBLK, sections 3.7 and 9.7
  - Updated column, section 6.2
  - Updated standards, section 7.0
  - Updated sections 8.2 and 10.3
  - Removed requirement for DoD IS to link to ICIS, section 10.7.3
  - Removed hardcopy requirement for manual integrations, sections 10.7.6 and 11.15.4.
  - Added matrix affecting retention times, section 11.1
  - Updated section 11.1.2

  - Added Data Review Checker (DRC), section 11.16.1
  - Updated Tables

- Revision 2 dated 26 July 2016
  - Added criteria to replace ICAL reagents opened frequently, section 7.7
  - Added criteria for checking ICAL older than 3 months, section 10.5.18
  - Updated example sequence, section 10.15
  - Added Chrom worklist instructions, section 11.15.1

- Revision 1 dated 16 January 2015
  - Added calibration points to example calibration in section 7.2.3.1.
  - Changed benzidine and PCP tailing factors to < 2. Section 10.4.5.
  - Changed resolution requirement to 50% in section 10.5.7.
  - Poor performers in ICV changed to 50-150% in section 10.5.15.
  - Changed CCV criteria for DOD to 20% for all reported analytes in section 10.6.3.
  - Changed table 9 to reflect 50% resolution requirement.
  - Changed table 9 to reflect proper tailing requirement.

- Revision 0, dated 07 February 2014 (initial revision using existing 8270C SOP (TA-MS-0313) updated to include 8270D requirements and remove non-applicable 8270C requirements)

Table 1: Current Compounds Applicable to Method

8270D Full Scan			
1,1'-Biphenyl	2-Fluorophenol	Atrazine	Di-n-octyl phthalate
1,2,4,5-Tetrachlorobenzene	2-Methylnaphthalene	Azobenzene	Fluoranthene
1,2,4-Trichlorobenzene	2-Methylphenol	Benzidine	Fluorene
1,2-Dichlorobenzene	2-Nitroaniline	Benzo[a]anthracene	Hexachlorobenzene
1,3-Dichlorobenzene	2-Nitrophenol	Benzo[a]pyrene	Hexachlorobutadiene
1,3-Dinitrobenzene	3 & 4 Methylphenol	Benzo[b]fluoranthene	Hexachlorocyclopentadiene
1,4-Dichlorobenzene	3,3'-Dichlorobenzidine	Benzo[g,h,i]perylene	Hexachloroethane
1,4-Dioxane	3-Nitroaniline	Benzo[k]fluoranthene	Hexadecane
1-Methylnaphthalene	4,4'-DDD	Benzofluoranthene	Indene
2,2'-oxybis[1-chloropropane]	4,4'-DDE	Benzoic acid	Indeno[1,2,3-cd]pyrene
2,3,4,6-Tetrachlorophenol	4,4'-DDT	Benzyl alcohol	Isophorone
2,4,5-Trichlorophenol	4,6-Dinitro-2-methylphenol	Bis(2-chloroethoxy)methane	Naphthalene
2,4,6-Trichlorophenol	4-Bromophenyl phenyl ether	Bis(2-chloroethyl)ether	n-Decane
2,4'-DDE	4-Chloro-3-methylphenol	Bis(2-ethylhexyl) phthalate	Nitrobenzene
2,4-Dichlorophenol	4-Chloroaniline	Butyl benzyl phthalate	N-Nitrosodimethylamine
2,4-Dimethylphenol	4-Chlorophenyl phenyl ether	Caprolactam	N-Nitrosodi-n-propylamine
2,4-Dinitrophenol	4-Nitroaniline	Carbazole	N-Nitrosodiphenylamine
2,4-Dinitrotoluene	4-Nitrophenol	Chrysene	n-Octadecane
2,6-Dichlorophenol	Acenaphthene	Dibenz(a,h)anthracene	Pentachlorophenol
2,6-Dinitrotoluene	Acenaphthylene	Dibenzofuran	Phenanthrene
2-Chloronaphthalene	Acetophenone	Diethyl phthalate	Phenol
2-Chlorophenol	Aniline	Dimethyl phthalate	Pyrene
2-Fluorobiphenyl	Anthracene	Di-n-butyl phthalate	Pyridine

8270D SIM		
1,3-Dinitrobenzene	Acenaphthylene	Hexachlorobenzene
1,4-Dioxane	Anthracene	Hexachlorobutadiene
1-Methylnaphthalene	Benzo[a]anthracene	Hexachlorocyclopentadiene
2,4,6-Trichlorophenol	Benzo[a]pyrene	Hexachloroethane
2,4-Dinitrophenol	Benzo[b]fluoranthene	Indeno[1,2,3-cd]pyrene
2,4-Dinitrotoluene	Benzo[g,h,i]perylene	Naphthalene
2,6-Dinitrotoluene	Benzo[k]fluoranthene	Nitrobenzene
2-Fluorobiphenyl	Benzofluoranthene	Nitrobenzene-d5
2-Fluorophenol	Bis(2-chloroethyl)ether	N-Nitrosodimethylamine
2-Methylnaphthalene	Chrysene	N-Nitrosodi-n-propylamine
3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene	Pentachlorophenol
4-Chloroaniline	Fluoranthene	Phenanthrene
Acenaphthene	Fluorene	Pyrene

Table 2: Suggested Instrument Conditions

Mass Range:	*35 - 550 amu for SCAN, select ions for SIM*
Scan Time:	*About 3 scan/sec*
Initial Column Temperature/Hold Time:	*45 ℃ hold 3 min*
Column Temperature Program:	*30 ℃/min to 280 ℃* *9 ℃/min to 325 ℃*
Final Column Temperature/Hold Time:	*325 ℃ hold for 2 min*
Injector Temperature:	*260 ℃*
Transfer Line Temperature:	*280 ℃*
Source Temperature:	*According to manufacturer's specifications*
Injector:	*Pulsed splitless*
Sample Volume:	*1.0 µl or 2.0 µl*
Carrier Gas:	*Helium at 3.3 mL/min.*

Current instrument conditions can be found noted in the maintenance logbook for each instrument.

Table 3: DFTPP Key Ions and Ion Abundance Criteria

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

Table 4: Surrogate Compounds

8270C Full Scan

Surrogate Compounds	Spiking Level, µg/mL in standard
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
Phenol-d5	100
2-Fluorophenol	100
2,4,6-Tribromophenol	100

Recovery limits for surrogates are generated from historical data and are maintained in the LIMS.

8270C SIM PAH

Surrogate Compounds	*Spiking Level, µg/mL in standard*
Terphenyl-d14	*100*
2,4,6-Tribromophenol[2]	*100*
2-methylnaphthalene-d10[1]	*100*
Fluoroanthene-d10[1]	*100*

Recovery limits for surrogates are generated from historical data and are maintained in the LIMS.

1. Included in standard mix, but not routinely evaluated for method 8270C SIM PAH list, non-DoD projects.

2. Included in standard mix, but not routinely evaluated for method 8270C, unless Pentachlorophenol or other associated compound is a target analyte.

8270C SIM Alternative Analyte List

Surrogate Compounds	*Spiking Level, µg/mL in standard*
Nitrobenzene-d5	*100*
2-Fluorobiphenyl	*100*
Terphenyl-d14	*100*
2-Fluorophenol	*100*
2,4,6-Tribromophenol	*100*
2-methylnaphthalene-d10[1]	*100*
Fluoroanthene-d10[1]	*100*

Recovery limits for surrogates are generated from historical data and are maintained in the LIMS.

1. Included in standard mix, but not routinely evaluated for method 8270C SIM PAH list, non-DoD projects.

Table 5: Table of Poor Performing Compounds*

2,3,4,6-Tetrachlorophenol	4-Nitrophenol
2,3,5,6-Tetrachlorophenol	Aniline
2,4-Dinitrophenol	Benzidine
3-Nitroaniline	Benzoic Acid
3,3' Dichlorobenzidine	Carbazole
4-Chloroaniline	N-Nitrosodimethylamine
4-Nitroaniline	

* - This is not a comprehensive list and is subject to change. Each project's target list should be evaluated for poor performers.

Table 6: Summary of QC Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
DFTPP Tune	Prior to ICAL and at the beginning of each 12-hour period	See Section 10.5	Retune instrument and verify. Rerun affected samples.
Breakdown Check	At the beginning of each 12-hour period and prior to analyzing samples.	Degradation ≤ 20% for DDT. Benzidine tailing < 2.0 and PCP tailing < 2.0. **For DoD:** Benzidine and PCP should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check. No samples can be run until degradation is acceptable.
Minimum 5-point Initial Calibration	Initial calibration prior to sample analysis	Option 1: RSD for each analyte ≤ 20% (<15% for DOD) Option 2: Linear regression r ≥ 0.990 **Option 2 for DOD:** Linear regression r ≥ 0.995. Option 3: Non linear regression r^2 ≥ 0.990 and 6 points must be used.	Terminate analysis; correct the problem; recalibrate. Problem must be corrected. No samples may be run until ICAL has passed.
ICV	Following initial calibration.	70-130% for non-DoD projects (e.g., 8270D HSL components); and 50-150% for poor performers **For DoD:** 80 - 120% recovery	Terminate analysis; correct the problem; recalibrate.
Relative Retention Times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL. Laboratory may update RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (e.g. column clipping).

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
CCV	Daily before sample analysis and every 12 hours of analysis time.	**For non-DoD projects:** 80-120% recovery for all 8270 standard compounds in Table 1 and surrogates; 50-150% recovery for 8270 poor performing compounds in Table 5. **For DoD/BP LaMP projects:** ; 1. %D/Drift for all standard target compounds in Table 1 and surrogates ≤ 20%D; 2. Closing CCV requires 50-150%D for all compounds.	Correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.
Internal Standards (IS) verification	Every field sample, standard, and QC sample	Retention time ± 30 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard. **For DOD:** Retention time must be + or – 10 seconds from the RT of the midpoint standard in the ICAL.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples while system was malfunctioning is mandatory.
Method Blank	One per batch of 20 field samples or fewer.	The result must be < RL or < 1/10 the amount measured in any sample or 1/10 the regulatory limit. **For DoD:** No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit. For common laboratory contaminants no analytes detected > RL.	Re-extract and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
LCS	One per batch of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain	See Section 9.4 for additional requirements.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		all analytes to be reported. Must be within acceptance criteria specified by DOD, if available. Otherwise, use in-house control limits.	
Surrogate	All field and QC samples.	Must be within laboratory control limits, **For DoD:** Must be within acceptance criteria specified by DOD, if available. Otherwise, use in-house control limits.	See Section 9.6 for additional requirements.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported. Must be within acceptance criteria specified by DOD, if available. Otherwise, use in-house control limits.	See Section 9.5 for additional requirements.

Table 7A: Characteristic Ions – SIM PAHs

SIM Group	Quantitation Ion	Qualifier Ions	Compound
1	136	108	Naphthalene-d8 (istd)
1	128	102, 127	Naphthalene
2	152	122	2-methylnaphthalene-d10 (surr)
2	142	141, 115	2-methylnaphthalene
2	142	141, 115	1-methylnaphthalene
3	152	151, 153	Acenaphthylene
3	164	162	Acenapthene-d10 (istd)
3	153	154, 152	Acenaphthene
4	166	165, 167	Fluorene
4	330	141	2,4,6-Tribromophenol (surr)
5	266	264, 268	Pentachlorophenol
5	188		Phenanthrene-d10 (istd)
5	178	179, 176	Phenanthrene
5	178	179, 176	Anthracene
6	212	106	Fluoranthene-d10
6	202	101, 203	Fluoranthene
6	202	101, 203	Pyrene
6	244	122	Terphenyl-d14 (surr)
7	228	229, 226	Benzo(a)anthracene
7	240	236	Chrysene-d12 (istd)
7	228	226, 229	Chrysene
8	252	253, 126	Benzo(b)fluoranthene
8	252	253, 126	Benzo(k)fluoranthene
8	252	253, 126	Benzo(a)pyrene
8	264	260	Perylene-d12 (istd)
9	276	138, 277	Indeno(1,2,3-cd)pyrene
9	278	276, 138	Dibenz(a,h)anthracene
9	276	138, 277	Benzo(g,h,i)perylene

Table 7B: Characteristic Ions – Alternative SIM Method

SIM Group	Quantitation Ion	Qualifier Ions	Compound
1	88	58, 43	1,4-Dioxane
1	74	42, 43	N-Nitrosodimethylamine
1	112	64	2-Fluorophenol (surr)
2	99	71	Phenol-d5 (surr)
2	93	63, 95	Bis(2-chloroethyl)ether
2	152	150	1,4-Dichlorobenzene-d4 (istd)
3	70	42, 130	N-Nitrosodi-n-propylamine
3	117	201, 199	Hexachloroethane
3	82	128	Nitrobenzene-d5 (surr)
3	77	123, 65	Nitrobenzene
4	136	108	Napthalene-d8 (istd)
4	127	129, 65	4-Chloroaniline
4	225	190, 118	Hexachlorobutadiene
5	152	122	2-methylnaphthalene-d10 (surr)
5	237	235, 272	Hexachlorocyclopentadiene
5	196	198, 200	2,4,6-Trichlorophenol
5	172	171	2-Fluorobiphenyl (surr)
6	168	122, 76	1,3-Dintrobenzene
6	165	89, 63	2,6-Dinitrotoluene
6	164	162	Acenaphthene-d10 (istd)
6	184	63, 154	2,4-Dinitrophenol
6	165	89, 63	2,4-Dinitrotoluene
7	330	332	2,4,6-Tribromophenol (surr)
7	284	142, 249	Hexachlorobenzene
7	266	264, 268	Pentachlorophenol
7	188	94	Phenanthrene-d10 (istd)
8	212	106	Fluoranthene-d10 (surr)
8	244	122	Terphenyl-d14 (surr)
9	252	254, 154	3,3'-Dichlorobenzidine
9	240	236	Chrysene-d12 (istd)
10	252	253, 126	Benzo(a)pyrene
10	264	260	Perylene-d12 (istd)
11	278	276, 138	Dibenz(a,h)anthracene

Table 8: 8270D Minimum RF criteria

Analyte	Minimum RF for initial and continuing calibration
1,2,4-Trichlorobenzene	0.01
Acenaphthene	0.9
2,4-Dinitrotoluene	0.2
Pyrene	0.6
N-Nitroso-di-n-propylamine	0.5
1,4-Dichlorobenzene	0.01
Pentachlorophenol	0.05
Phenol	0.8
2-Chlorophenol	0.8
4-Chloro-3-methylphenol	0.2
N-Nitrosodimethylamine	0.01
Bis(2-chloroethyl)ether	0.7
n-Decane	0.01
1,3-Dichlorobenzene	0.01
Benzyl alcohol	0.01
1,2-Dichlorobenzene	0.01
2-Methylphenol	0.7
2,2'-oxybis[1-chloropropane]	0.01
3 & 4-Methylphenol	0.6
Hexachloroethane	0.3
Nitrobenzene	0.2
Isophorone	0.4
2-Nitrophenol	0.1
2,4-Dimethylphenol	0.2
Benzoic Acid	0.01
Bis(2-chloroethoxy)methane	0.3
2,4-Dichlorophenol	0.2
Naphthalene	0.7
4-Chloroaniline	0.01
Acetophenone	0.01
Hexachlorobutadiene	0.01
4-Nitrophenol	0.01
2-Methylnaphthalene	0.4
1-Methylnaphthalene	0.4
Hexachlorocyclopentadiene	0.05
2,4,6-Trichlorophenol	0.2

2,4,5-Trichlorophenol	0.2
2-Chloronaphthalene	0.8
2-Nitroaniline	0.01
Dimethyl phthalate	0.01
2,6-Dinitrotoluene	0.2
Acenaphthylene	0.9
3-Nitroaniline	0.01
2,4-Dinitrophenol	0.01
Dibenzofuran	0.8
2,3,4,6-Tetrachlorophenol	0.01
Diethyl phthalate	0.01
4-Chlorophenyl phenyl ether	0.4
4-Nitroaniline	0.01
Fluorene	0.9
4,6-Dinitro-2-methylphenol	0.01
N-Nitrososdiphenylamine	0.01
Azobenzene	0.01
4-Bromophenyl phenyl ether	0.1
Hexachlorobenzene	0.1
n-Octadecane	0.01
Phenanthrene	0.7
Anthracene	0.7
Di-n-butyl phthalate	0.01
Fluoranthene	0.6
Butyl benzyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
Bis(2-ethylhexyl) phthalate	0.01
Benzo(a)anthracene	0.8
Chrysene	0.7
Di-n-octyl phthalate	0.01
Benzo(b)fluoranthene	0.7
Benzo(k)fluranthene	0.7
Benzo(a)pyrene	0.7
Indeno(1,2,3-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5
Carbazole	0.01

Attachment 1: Example Internal Standard Evaluation Custom Report

FORM VIII
GC/MS SEMI VOA INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: TestAmerica Tacoma Job No.: 580-12197-1

SDG No.:

Sample No.: CCVIS 580-39026/2 Date Analyzed: 12/10/2008 13:19

Instrument ID: TAC040 GC Column: ZB-5MS ID: 0.25(mm)

Lab File ID (Standard): ak018739.D Heated Purge: (Y/N) N

		DCB		NPT		ACN	
		AREA #	RT #	AREA #	RT #	AREA #	RT #
12 HOUR STD		11172	3.20	17716	4.18	11176	5.59
UPPER LIMIT							
LOWER LIMIT							
LAB SAMPLE ID	CLIENT SAMPLE ID						
MB 580-38946/1-A		7654	3.20	18328	4.18	11486	5.59
LCS 580-38946/2-A		8484	3.20	19108	4.18	11938	5.59
580-12197-3	08FTW336B-32	7980	3.20	19016	4.18	11411	5.59
580-12197-3 MS	08FTW336B-32 MS	7765	3.20	17983	4.18	11310	5.59
580-12197-3 MSD	08FTW336B-32 MSD	7232	3.20	18301	4.18	11655	5.59

DCB = 1,4-Dichlorobenzene-d4
NPT = Naphthalene-d8
ACN = Acenaphthene-d10

Area Upper Limit = 200% of Internal Standard Area
Area Lower Limit = 50% of Internal Standard Area

Column used to flag values outside QC limits

FORM VIII 8270C

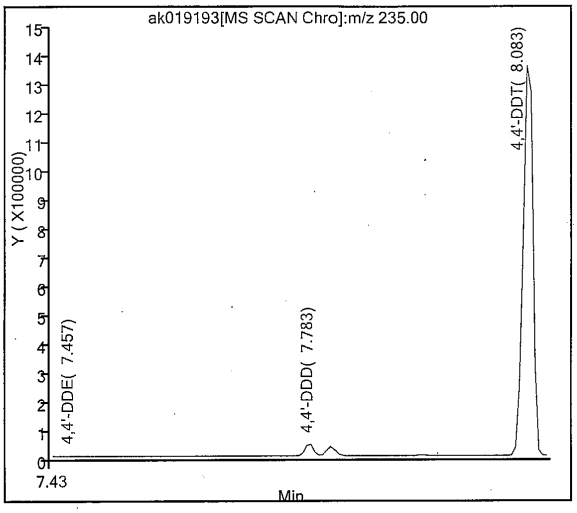
Attachment 2: Example Breakdown Evaluation Custom Reports

Report Date: 01-Apr-2009 15:08:23 Chrom Revision: 1.0.2009.312

Preliminary Report
Breakdown Report

Data File:	\\TACSVR5\Chromdata\TAC040\20090401-1.b\ak019193.D		
Inj Date:	01-Apr-2009 07:56:30	Limit Group:	8270C Standard
Lims Batch ID:	42046	Lims Sample ID:	1
Client ID:		Instrument:	TAC040
Sample Info:	dftpp		
Sample Amount:	1.00 uL	Operator:	RBF

93 4,4'-DDT, Detector: MS SCAN



SW-846 Method

%Breakdown =
(Area Breakdown Cpnds/
Total Area Breakdown Cpnds) * 100

93 4,4'-DDT Area = 1276732
89 4,4'-DDE Area = 2208
91 4,4'-DDD Area = 33705

%Breakdown: 2.74%, Max Limit: 20.00%
Passed

Attachment 3: Example Tailing Evaluation Custom Reports

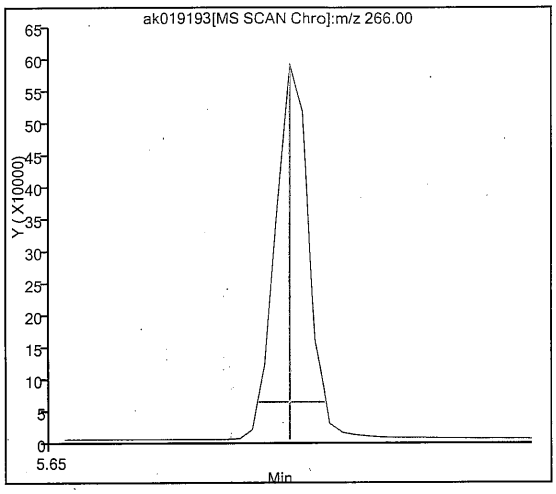
Report Date: 01-Apr-2009 15:09:32 Chrom Revision: 1.0.2009.312

Preliminary Report

Peak Tailing Report

Data File: \\TACSVR5\Chromdata\TAC040\20090401-1.b\ak019193.D
Inj Date: 01-Apr-2009 07:56:30 Limit Group: 8270C Standard
Lims Batch ID: 42046 Lims Sample ID: 1
Client ID: Instrument: TAC040
Sample Info: dftpp
Sample Amount: 1.00 uL Operator: RBF

60 Pentachlorophenol_T, Detector: MS SCAN

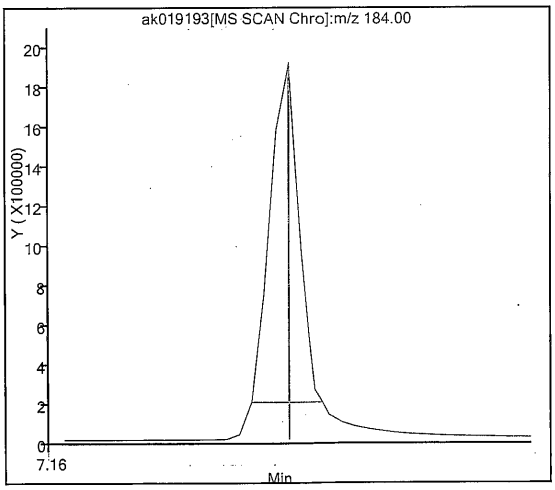


Peak Tailing Factor =
BackWidth/FrontWidth @ 10% Peak Height

Back Width = 0.015 (min.)
Front Width = 0.014 (min.)

Tailing Factor = 1.1, Max. Tailing < 5.00
Passed

85 Benzidine_T, Detector: MS SCAN



Peak Tailing Factor =
BackWidth/FrontWidth @ 10% Peak Height

Back Width = 0.013 (min.)
Front Width = 0.016 (min.)

Tailing Factor = 0.8, Max. Tailing < 3.00
Passed

APPENDIX A

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure	**Check mass calibration (PFTBA or FC-43).**	**Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.**	**Check vacuum, relays, gas pressures, and flows.**	**Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.**
Check temperatures of injector, detector. Verify temperature programs.		**Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.**		**Change the oil in the mechanical rough pump.**
Check inlets, septa.		**Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.**		**Relubricate the turbomolecular pump-bearing wick.**
Check baseline level.		**Repair/replace jet separator.**		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		**Replace filaments when both filaments burn out or performance indicates the need for replacement.**		

APPENDIX A (continued)
Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	**Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).**
Check temperatures of injectors and detectors. Verify temperature programs.	**Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.**
Check inlets, septa. **Clean injector port.**	**Replace septa.**
Check baseline level.	**Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).**
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	**Repair or replace flow controller if constant gas flow cannot be maintained.**
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Determination of Volatile Organic Compounds and Total Purgeable Petroleum Hydrocarbons by GC/MS [Methods 8260B, 8260C, 624]

Approvals

Signatures on File	
Isaac Hooper Date	Manjit Nijjar Date
Volatiles Department Manager	Health & Safety Manager / Coordinator
Terri Torres Date	Dennis Bean Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Tables 1, 2, and 3.

1.2 This SOP is applicable to Method 8260B and 8260C. Appendix A presents modifications to the procedures in the main SOP that are necessary for analysis of wastewater by Method 624 (CWA compliance testing). It is important that the differences among these methods are carefully observed.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 1 to *100* µg/L for 8260B and 8260C waters, 1 to *100* µg/kg for low-level soils, and 8 to 6,000 µg/kg for *medium*-level soils. The working range for Method 624 (5 mL purge) is 1-*100* µg/L.

1.5 Reporting limits *can be located in TALS > Global Method Data > Methods (select method) > Limits View > LT Code (select "RL") > Select Matrix*.

1.6 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS/*LCSD)*.

1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate; sampled directly into pre-tarred VOA vials which contain 5mL reagent free water, a magnetic stir bar, and are immediately frozen; or collected in a suitable container to be transferred in total or by aliquot to a VOA vial and purged directly.

2.3 In the purge-and-trap process, an inert gas (generally *Helium*) is bubbled through the solution at ambient temperature and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 Definitions

3.1 Both SW-846 (RCRA) and drinking water (SDWA) terminology are used in this section for cross-reference purposes. Elsewhere in the SOP, the SW-846 terminology is used exclusively.

3.2 Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each 4-bromofluorobenzene (BFB) analysis will normally start a new batch. Batches for high-level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

The Quality Control batch must contain a matrix spike/spike duplicate pair (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. If there is insufficient sample to perform the MS/MSD, a duplicate LCS is used to establish batch precision when requested by the client. Refer to SOP TA-QA-0620 for further details of the batch definition.

3.3 Method Blank (MB) or Laboratory Reagent Blank (LRB)

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data. Sparged water or water that has been boiled then cooled to ambient temperature is used as the blank medium for water batches and muffled Ottawa Sand for soil batches. Prepared (muffled at *400*C for at least *4* hours) batches of Ottawa sand are tracked using the reagent data base in the Laboratory Information Management System (known as TALS) and are at the time of the writing of this SOP named with the following convention: VoaSand_XXXXX.

3.4 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)

A blank matrix (reagent water or muffled Ottawa Sand) is spiked with the analytes of interest and is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.5 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD) or Laboratory Fortified Sample Matrix (LFM)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.7 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift (%D) for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.8 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.9 Initial Calibration Verification (ICV) or Quality Control Sample (QCS)

The ICV is a second-source calibration verification standard. The QCS is reagent water or an environmental sample that is fortified with target analytes at known concentrations. This too is a second-source standard, i.e., different than the source of calibration standards.

3.10 Continuing Calibration Verification (CCV) or Laboratory Performance Check Solution (LPC)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, boiled and cooled to ambient or sparged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly Methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

Note: Due to the large number of analytes analyzed for in this method, some with higher boiling points are considered Semi-Volatile analytes. It may be necessary to evaluate for cross-contamination at levels above 10 ug/L for Naphthalene and 1,2,3-Trichlorobenzene; above 20 ug/L for 2-Ethyl-1-hexanol, n-Butylbenzene and Hexachlorobutadiene; above 50 ug/L for tert-Butylbenzene, sec-Butylbenzene, 4-Isopropyltoluene, 1,2,4-Trichlorobenzene and Ethylbenzene; above 100 ug/L for Methacrylonitrile, 1,3,5-Trichlorobenzene, 1,2-Dibromo-3-Chloropropane and Toluene, and all other analytes should be evaluated for potential cross-contamination above a detected concentration of 150 ug/L or more. (These concentrations are based on a carry-over study conducted by the laboratory detecting cross-contamination above ½ the RL). It may, therefore, be necessary to run an instrument rinse after laboratory spiked samples, such as high calibration levels, CCVs, LCS and MS to ensure no cross contamination occurs. **All client samples are also evaluated using the same criteria.**

4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used *and must also be added to the Method Blank (MB).*

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 The autosampler, purge and trap, gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 Cut resistant gloves or a protective cloth must be used when opening voa vials.

5.1.5 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.
- Gas Chromatographic Column used for 8260:

  : 60 m X 0.25mm ID DB-VRX with 1.4 µm film thickness.

  Note: Other columns may be used. The serial number of the column used is documented in the instrument maintenance logbook.
- Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria.
- GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.
- Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

- Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.
- Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax/Silica gel/Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.
- Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.
- Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.2 Computer hardware and software

- Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.
- LIMS system: TALS version 1.0 or higher.
- Data acquisition system: Agilent (Hewlett Packard) ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.
- Data processing: Chrom version 1.2 or higher. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.3 Supplies

- Microsyringes: 0.5 µL gas tight and larger, 0.006-inch ID needle
- Balance: Top-loading balance capable of weighing 0.1 g. The balance used for sample preparation is calibrated daily by a designated primary analyst (a back-up analyst is also assigned should the primary be unavailable). The analyst must perform this check according to SOP TA-QA-0014. It is also the responsibility of any analyst performing work on the balance to check the Balance logbook to determine if the daily calibration check has been completed, before beginning work.
- Scintillation Vials: 20 mL with screw caps.
- Volumetric flasks: 10 mL to *200* mL, class A with ground-glass or Teflon ® stoppers.
- Spatula: Stainless steel.
- Disposable pipettes: Pasteur.
- pH paper: Wide range (0-14) and narrow range (0-2.5).
- Helium: Ultra high purity, gr. 5, 99.999%.
- Nitrogen: Ultra high purity, from cylinders or gas generators, may be used as an alternative to helium for purge gas.

Note: The use of Nitrogen as a purge gas is not allowed for analysis of VOA contaminants in drinking waters.

- Compressed air: Used for instrument pneumatics.

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Reagents

7.2.1 Methanol: Purge and Trap Grade, High Purity

7.2.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas for a minimum of 1 hour or boiling and cooling to ambient temperature prior to use. Other methods of preparing reagent water are acceptable.

7.3 Standards

7.3.1 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturer's expiration date.

7.3.1.1 Purchased standards are stored at the manufacturer's specifications (i.e. ambient, freezer, refrigerator). Standards prepared from these purchased standards are stored in the freezer.

7.3.2 Calibration Stock Standard Solutions: Components of stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at ≤-10°C. Each *month a new standard is prepared.* Note: *Standards* may be prepared on a more frequent basis based on analyst observed signs of degradation.

7.3.3 Calibration Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. Generally, an analysis of all individual compounds meeting the method criteria will suffice, however a continual failure of CCC compounds may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem (two CCVs in a row fail), then a new initial calibration must be performed.

7.3.4 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

Likewise, *medium l*evel methanolic calibration standards are prepared in reagent water with a matrix matched methanol concentration of one fortieth P&T methanol using the secondary dilution standards and must be prepared daily.

7.3.5 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Table 5 for internal standard components.

7.3.6 Surrogate Standards: Refer to Table 6 for surrogate standard components and spiking levels.

7.3.7 Laboratory Control Sample Spiking Solutions: Refer to Tables 7 and 7a for LCS components and spiking levels.

7.3.8 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Tables 7 and 7a.

7.3.9 Tuning Standard: A standard is made up that will deliver *a maximum* 50ng of 4-Bromofluorobenzene on column upon injection.

7.4 As soon as standard preparations are completed, the *working* standards must be returned to the freezer.

7.5 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. The holding time for acid-preserved samples is 14 days from sample collection. For compliance with Method 624, 8260B and 8260C, unpreserved samples must be tested within 7 days of collection.

8.2 There are two exceptions to the information provided in Section 8.1 above:

8.2.1 2-Chloroethyl vinyl ether, acrolein, and acrylonitrile are hydrolyzed in the presence of acid. For samples collected for analysis of this compound, a separate vial without acid should be recommended.

8.3 Soils

8.3.1 Approved sampling containers for Method 5035 are EnCores, VOAs (with or without water and stir bar), and VOAs preserved with methanol or with sodium bisulfate

8.3.2 The holding time for sodium bisulfate and methanolic preserved samples is 14 days.

8.3.3 Soil collected using the EnCore™ sampler must be preserved in the laboratory within 48 hours of sampling. At specific client request, if the data is to be reported as 5030, the hold time for preservation and analysis is 14 days from collection. The holding time for EnCore™ samples varies based on client specifications and can be 48 hours, 7 days, or 14 days, see the table in Section 8.4.

8.3.4 The holding time for VOAs (with or without water and stir bar) varies based on client specifications and requirements and can be 48 hours, 7 days, or 14 days, see the table in Section 8.4.

8.4 Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Three 40-mL VOA vials	40 mLs	HCl, pH < 2; Cool 0-6°C	14 Days	40 CFR Part 136.3 / 5030
Waters	Three 40-mL VOA vials	40 mLs	Cool 0-6℃	7 Days	40 CFR Part 136.3 / 5030
Soils	Three Encore Samplers	5 grams	Cool 0-6℃ or -10 to -20℃	48 Hrs for Preservation or 14 Days	5035A
Soils	Three VOA vials	5 grams	Sodium bisulfate Cool 0-6℃	14 Days	5035A
Soils	40 ml VOA vial or 4oz septa top jar	10 grams or 25 grams	Methanol Cool 0-6℃	14 Days	5035A
Soils	Three 40-mL VOA vials	5 grams	With or without DI Water -10 to -20℃	14 Days	5035A

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass Jar	10 grams	Cool 0-6°C	5035A:48 Hrs for Preservation 14 Days 5030: 14 Days	5035A or 5030C
Waste	Glass Jar	10 grams	Cool 0-6°C	14 Days	5030C

[1] Inclusive of preparation and analysis.

8.5 Aqueous samples are stored in three 40 ml glass VOA vials with Teflon lined septa at 0-6°C, with minimal headspace. If a bubble is present and it is less than 6 mm in diameter, analysis may continue with the appropriate NCM added. If headspace exceeds this amount, then a non-conformance memo must be written and in some cases client approval requested to continue analysis.

8.6 Soil Sample Collection for *Medium*-Level Analysis using Field Methanol Preservation

8.6.1 A pre-tared four ounce volatile soil jar with an accompanying VOA vial containing 25ml of a methanol/surrogate solution containing the surrogate TFT is sent out for each sample required when the sampling occurs in the state of Alaska. Otherwise a pre-tarred VOA vial containing 10 mls of methanol is sent out for each sample required. In addition the appropriate amount of trip blanks are also sent out. All bottles sent to the field are labeled with the tare weight and lot number of the methanol/surrogate solution or methanol.

8.6.2 Field samples are collected *in a 1:1 ratio, (grams of sample:mL of MeOH). Alaska samples are* placed in the *collection container* with septa lined lid *prior to adding* one VOA vial containing 25 mls of methanol/surrogate solution. *For all other methods* ten gram field samples are collected by adding an appropriate amount of sample to a 40 mL VOA vial *already c*ontaining 10 mL of methanol.

8.7 Soil Sample Collection for *Medium*-Level Analysis using EnCore™ or TerraCore™ samplers.

8.7.1 If the sample is collected a sample in an EnCore™ sampler a minimum of one EnCores should be provided to the lab. If the samples are collected using the TerraCore sampler a minimum of one 40-ml VOA vial with 5 grams of soil. Samples must be received prior to 48 hours from sampling in order to be frozen or extracted in methanol. Following shipment back to the laboratory, the soil is preserved in methanol.

8.8 Soil Sample Collection for *Medium*-Level Analysis *of U*npreserved *Soil*

8.8.1 When specifically requested by a client, unpreserved soils packed into glass jars or brass tubes may be accepted, subsampled and methanol preserved in the laboratory. These samples have a hold time of 48 hours from sample collection to sub-sampled and preserved if following Method 5035. Otherwise if following Method 5030 the holding time is 14 days to analysis.

8.9 Soil Sample Collection for Low-Level Procedure

8.9.1 Samples may be collected in 5g EnCore™ sampling device (it is recommended that a minimum of two 5g EnCore™ samples are collected, but three are preferred). Soil samples collected in a 5g EnCore™ sampling device and returned to the laboratory are extruded into a VOA vial with stir bar and frozen upon receipt or extruded into a scintillation vial with methanol and refrigerated upon receipt.

8.9.2 Samples may be collected in vendor purchased pre-tared VOA vials with or without 10 mL of DI Water with a magnetic stir bar. Samples collected in pre-tared VOA vials are stored at -10 to -20℃ in the volatile laboratory soils fr eezer until sample analysis.

8.9.3 Samples may be collected in vendor purchased pre-tared VOA vials containing 5 or 10 mL of 20% Sodium Bisulfate solution with a magnetic stir bar. Due to the exhibited potential of positive interferences in the purchased prepared vials (most notably ketones and BTEX

compounds). Samples collected in pre-tared Sodium Bisulfate preserved sample vials are stored at 0-6℃ in the volatile laboratory soils re frigerator until sample analysis.

8.9.3.1 Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, samples should be taken in a 5g EnCore™ sampling device, and stored at ≤–10℃ until analysis.

8.9.3.2 If client specifications require field preservation, samples may be collected in pre-tared VOA vials containing a magnetic stir bar and 5 mL of reagent water. Sample collected in this manner must be received and frozen by the laboratory within 48 hours of sampling. Samples stored in this manner **MUST** be frozen on their sides to minimize possible breakage of the sample container due to expansion of water as it freezes.

8.10 A refrigerator or freezer blank is stored in each refrigerator or freezer with the samples. This is analyzed at minimum every 14 days, but may be analyzed more frequently as needed (see SOP TA-QA-0616). The refrigerator or freezer blank should be run immediately after the method blank.

8.11 Percent Moisture Correction for Soils

A percent moisture correction may be performed on soil samples to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect, as required by the client. Percent moisture must be determined if results will be reported as dry weight and percent moisture correction to be performed; refer to SOP TA-WC-0125 for determination of percent moisture. For all methanolic samples with a % moisture of greater than 10%, the following formula is used to determine the corrected final volume:

Corrected FV = ((g of sample * % Moisture/100) + mL of Methanol) * 40

(Also noted in section 12.9)

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in the TestAmerica Seattle QAM.

9.1.2 Specific QC requirements for Federal programs, e.g., USACE and Navy projects, are described in DoD QSM v5*.1* or the latest promulgated version.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and may also come in the form of email or written notifications distributed at "project kick off" meetings.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or

in the same sequence. The method blank must be run on each instrument and in each analytical batch.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards and before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is muffled Ottawa sand. For *medium*-level volatiles, the method blank consists of 10 mL of reagent grade methanol and ten grams of muffled Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: For DoD/LaMP projects the method blank must not contain any analyte of interest at or above one-half the reporting limit or above 1/10 the measured concentration of the analyte in the associated samples or 1/10 the regulatory limits, whichever is greater. For DoD projects, when written approval is received (method notes will contain “2CLC” or “Std Var App” to indicate approval has been received), the method blank must not contain any common laboratory contaminants above the reporting limit. Contamination up to the reporting limit is allowed for non DoD/LaMP projects or at or above 1/10 of the measured concentration of that analyte in the associated samples, whichever is higher.

The method blank must have acceptable surrogate recoveries.

Corrective Actions: For DoD projects, if the analyte is a common laboratory contaminant (i.e., acetone, 2-butanone, carbon disulfide and methylene chloride), and written approval has been received, the data may be reported with qualifiers if the concentration of the analyte is less than the reporting limit. For non-DoD if the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone, ethyl ether, Acetonitrile and hexane) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than the RL (less than one half the RL for DoD clients) in the samples associated with an unacceptable method blank, the data may be reported with qualifiers for non-DoD clients. For DoD clients the data may only be reported if written approval has been received.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all affected analytes in the associated samples are flagged as required by the project, and appropriate comments may be made in a narrative to provide further documentation.

9.4 Surrogates

Every sample, blank (including instrument blanks), and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that

recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 6.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean or as defined by project or program requirements. Surrogate recovery limits are updated at a fixed frequency by QA and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

NOTE: For LaMP client samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the LaMP client representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

9.5 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard and the method blank, and normally before any samples. The LCS contains all of the analytes of interest (see Table 7a) and must contain the same analytes as the matrix spike.

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data or based on project/program limits. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system

is in control and validates system performance for the samples in the associated batch. Recovery limits are updated at a set frequency by QA and are stored in the LIMS.

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAC. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

For non-DoD projects, if the LCS recovery is high and there are no detections in the associated samples for the affected analytes the data may be reported with qualifiers. For DoD projects the data may only be reported with qualifiers if approval has been received in writing (method notes will contain “3HR” or “Std Var App” to indicate approval has been received).

Note: For DOD projects, all exceedances of LCS Control Limits, are subject to corrective action. Therefore, all instances of LCS failures including the high bias but not detected in the associated samples scenario, must be investigated. For example and as noted above, the randomness of these failures can be evaluated or the spike solution can be re-verified. When the source of the problem is identified, corrective action is taken or variance from the QSM is requested.

Corrective Actions:

If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records (via NCMs and the case narrative) and in the final report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Tables 7 and 7a. The selection of the spike solution is dependent on client program requirements. The matrix spike/duplicate must be analyzed at the same base dilution as the unspiked sample, even if the matrix spike compounds will be diluted out. Dilutions (beyond the base dilution if necessary) of MS/MSD analyses are not required unless there are specific client instructions to do so. If necessary, this requirement will be passed to the laboratory through the PM by means of the mechanisms described in section 9.1.3 of this SOP.

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated at a regular frequency by QA and are stored in the LIMS.

Corrective Actions: If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/matrix spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch, except in cases where a high bias is indicated and no target is detected above the reporting limit in any associated sample.

If an MS/MSD is not possible due to limited sample, then a LCS duplicate (LCSD) should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 If batch QC samples or trip blanks are re-analyzed to confirm a recovery or result, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (i.e. no internal standard added, bent autosampler needle, etc).

9.8 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

10.1 Samples scheduled for EPA 624 will be analyzed separately (different ICAL and sequence) from samples scheduled for EPA 8260B or 8260C. Refer to appendix A for Modifications for Method 624.

10.2 One time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance

Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.0 Calibration

11.1 Summary

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of a separate calibration is required for low level water.

11.2 Recommended Instrument Conditions

11.2.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 seconds/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute (± 5 mL/min)
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

11.2.2 Gas Chromatograph Temperature Program

The temperature programs vary with the column type and instrumentation used. The GC run program on each instrument should be optimized so that each peak is broad enough to accommodate at least 5 scans across the peak (not counting the scans at the baseline start and end of the peak). The actual individual method parameters used are stored in each individual instrument methods folder on the network and can be referenced there.

11.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed below and in Table 8 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB. It is critical to accurately estimate the number of samples that can be analyzed within the 12-hour window. When a tune isn't analyzed every 12-hours (i.e., samples are analyzed outside of the 12-hour window), the event must be documented in a non-conformance memo and corrective *action m*ust be taken. Whenever feasible, samples that were analyzed outside of the 12-hour window will be re-analyzed within a new 12-hour window. When reanalysis is not feasible, results for the affected samples can only be reported if it's technically justified (e.g., subsequent tune passes), the data has been qualified, and it's been authorized and accepted by the client. The BFB may be taken from a specified BFB Tune injection or from the CCVIS. In the case of a calibration sequence, a specified BFB Tune must be injected prior to the injection of the first calibration standard. If an acceptable tune is not achieved, the autosampler prepares another tune standard by adding BFB to either the CCVIS or an instrument blank. The autotune process is repeated once. If the subsequent tune attempt fails, one or more of the corrective actions suggested in the TestAmerica, Inc. corporate tune policy, CA-Q-QM-002 are *to be* attempted.

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	50 to 120 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

11.4 Initial Calibration

11.4.1 A series of five or more initial calibration standards is prepared and analyzed for the target compounds. Nominal calibration levels for a standard level water purge, low level soil purge and *medium* level methanolic extract purge are 0.2, 0.4, 1, 2, 5, 10, 20, 50, 100 and 150 µg/L. Low level waters curves are prepared at 0.02, 0.05, 0.10, 0.20, 0.40, 1, 5, 10, 25, 40, and *50* ug/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Table 4 shows the calibration levels for each analysis. The purge volume is *5 mL for standard level waters and* 15 mL *for low level waters*. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements. Calibration levels may also vary based on analyst discretion in so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the lowest point on the curve is at or below the current TestAmerica Seattle reporting limit.

11.4.2 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

11.4.3 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

11.4.4 Rejection of Calibration Points

Calibration levels below the reporting limit may be removed provided that the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves), and the lowest standard is at or below the TestAmerica Seattle reporting limit.

High point calibration levels may also be removed so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the midpoint of the curve (the ICIS) is not the highest point of the calibration range.

Generally, it is NOT acceptable to remove mid-points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the maintenance logbook. Then the calibration standards must be reanalyzed. If, however, there is documented evidence of a problem with a calibration

point (e.g. misinjection, poorly sealed vial, etc…) then one point might be rejected, but it is recommended to re-calibrate.

Refer to Corporate SOP GA-Q-S-005, Calibration Curves, for further details.

11.4.5 Internal Standards

Internal standard calibration is used. The internal standards are listed in Table 5. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Corporate SOP CA-Q-S-005, for calculation of response factor and other related algorithms.

11.4.6 Calibration Check Compounds (CCC), 8260B

For 8260B the % RSD of the calibration check compounds (CCC) must be less than or equal to 30% even in cases where a first or second order regression is used for the calibration curve. Refer to Table 10. If the %RSD exceeds 30% for any CCC, the system must be evaluated (e.g. maintenance and accuracy of calibration standards) and the calibration re-run.

11.4.7 System Performance Check Compounds (SPCC), 8260B

The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. For 8260B the five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 9 for the SPCC compounds and required minimum response factors. If the minimum response factors are not met for any CCC, the system must be evaluated (e.g. maintenance and accuracy of calibration standards) and the calibration re-run.

11.4.8 For 8260C the most common target analytes are checked for a minimum response factor for each calibration level. See Table 14 for the compound list and minimum relative response factor criteria. 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 this criterion. Compounds which commonly fail the criteria have been studied for an appropriate RF factors using data over a period of 3 months from all instruments used for 8260C analysis. These compounds have RF criteria based on their average RF over the time period and subtracting one standard deviation in order to include all acceptable data. For any analyte non-detect associated with a calibration that fails the minimum response factor criteria there must be a demonstration of adequate sensitivity at the quantitation limit. This is achieved by the successful analysis of a CCVL (CCV at the reporting limit) in the same analytical batch. The criterion for the CCVL is detection only but the standard qualitative identification criteria in the method must be met.

11.4.9 If all of the %RSD values in the calibration are ≤ 15% for 8260B, then all analytes may use average response factor for calibration. If all of the %RSD values in the calibration are ≤ 20% for 8260C, then all analytes may use average response factor for calibration. For analytes that fail the RSD criteria, use linear or quadratic curve.

11.4.10 If the software in use is capable of routinely reporting curve coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD ≤ 15% (8260B or DoD) or ≤ 20% (8260C) for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve, then the appropriate curve should be used for quantitation. The correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r^2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.11 If the software in use is capable of routinely reporting curve coefficients for data, then calibration on a curve must be used for all analytes with %RSD > 15% (8260B) or %RSD > 20% (8260C). The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r^2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.12 For 8260C no more than 10% of compounds can fail the 20% RSD/0.990 correlation coefficient requirement. Any individual analyte result that fails the 20%RSD/0.990 correlation coefficient requirement must be flagged or narrated as an estimated concentration.

11.4.13 For any analyte non-detect associated with a calibration that fails the 20%RSD/0.990 correlation criteria there must be a demonstration of adequate sensitivity at the quantitation limit. This is achieved by the successful analysis of a CCVL (CCV at the reporting limit) in the same analytical batch. The criterion for the CCVL is detection only but the standard qualitative identification criteria in the method must be met.

11.4.14 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

11.4.15 Initial Calibration Verification (ICV)

Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. The ICV is generally run at 20 ug/L for standard level water, low-level soil and methanol preserved soil, and 10 ug/L for low level water curves. As the ICV concentration must not be equal to or greater than the highest calibration level, other ICV levels than those previously listed may be used or multiple levels of ICV may be needed to validate all compounds in the initial calibration curve.

For DoD and LaMP projects each target compound in the ICV must be <20% drift when compared to the initial calibration. The same criteria apply to BTEX and oxygenate compounds in the LaMP program. For 8260B non-DoD and non-LaMP projects, all compounds must be <40% drift when compared to the initial calibration, except for poorly performing compounds listed in Table 11, which must be <55% drift. For 8260C non-DoD and non-LaMP projects, all compounds must be <30% drift when compared to the initial calibration, except for poorly performing compounds listed in Table 11, which must be <55% drift.

For DoD projects analyses may continue if the drift is <30% for identified poor performing analytes with written approval from the client. (Method comment 4PP). See table 11 for the list of indentified poor performers.

For non-DoD and LaMP projects analyses may continue for those analytes that fail the criteria with approval from the client and an understanding that these results would be considered estimates and could be used for screening purposes.

Corrective Action: If the % drift falls outside acceptance criteria, assess the system for possible problems (standard degradation, etc.), re-prepare the verification standard and re-analyze. If the second ICV also fails, corrective action is required (e.g. system maintenance, re-preparing intermediate standards, etc.) and the calibration must be re-prepared and re-analyzed. An acceptable ICV must be achieved before sample analysis. No samples may be run until calibration has been verified. Analytes which do not meet the ICV % drift criteria will be removed from the calibration in the Chrom chromatography software for the method in which the analyte did not meet the method criteria to prevent the reporting of analytes using a deficient calibration curve.

11.4.16 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 11.5.

11.5 Continuing Calibration

11.5.1 The initial calibration must be verified every twelve hours. For DoD projects the calibration must also be verified with a closing continuing calibration standard (CCVC) at the end of each analytical sequence.

11.5.2 Continuing calibration begins with analysis of BFB as described in Section 11.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 6 calibration standard is generally used as the CCV and CCVC.

11.5.3 For 8260B non-DoD projects the RF data from the standards are compared with the initial multi-point calibration to determine the percent drift of the CCC and target compounds. The % drift limits of CCC, target and surrogate compounds are summarized in Table 12.

11.5.3.1 If not all of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 10) must be agreed with the client.

11.5.3.2 Non CCC target compounds that exceed the specified limits for Drift or Difference should be flagged.

11.5.3.3 For sub lists having less then 10 target analytes, the % drift of difference should be <20% before analysis proceeds.

11.5.4 For 8260B (both DoD and non-DoD), the SPCCs are also monitored. The SPCCs must meet the criteria described in Table 9.

11.5.5 For 8260C non-DoD projects, the percent difference or drift (%D) of target and surrogate compounds must be within ± 20% for 80% of the target compounds.

11.5.6 For DoD and LaMP projects, the percent difference or drift (%D) of target and surrogate compounds must be within ± 20% for the opening CCV. The same criteria apply to BTEX, oxygenate and surrogate compounds in the LaMP program.

11.5.7 For DoD projects the percent difference or drift (%D) of target and surrogate compounds must be within ± 50% for the closing CCV (CCVC).

11.5.8 For non-DoD projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds (0.5 min) when compared to the most recent multi-point calibration. For DoD projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 10 seconds when compared to the most recent multi-point calibration. The 30 second criteria may only be used for DoD projects if prior written approval is received (method notes will contain “8ISRT” or “Std Var App” to indicate approval has been received). The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent multi-point calibration.

11.5.9 If CCC, SPCC, target and/or surrogate compounds do not meet the criteria in Sections 11.5.3 through 11.5.8, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration. For non-DoD and LaMP, if two CCVs in a row fail, a new initial calibration must be performed. For DoD two additional consecutive CCVs must be run immediately. If both pass, samples may be reported without reanalysis. If either fails corrective action and re-calibration is required.

Corrective Actions:

For non-DoD projects if the CCV recoveries of the CCC, target or surrogates compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias. For DoD projects, if the CCV recoveries for target compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified

and reported as the system has shown a potentially high bias only if prior approval has been received in writing (method notes will contain "3HR" or "Std Var App" to indicate approval has been received). For all other cases, results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

11.5.10 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

11.6 Sample Preparation

Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014

11.6.1 Sample extraction for *Medium*-Level Analysis using in house extraction.

11.6.1.1 When extracting the sample, extrude (for 5 g EnCore™) or weigh (for 10 g in house soil extraction) the sample into a tared 20 mL scintillation vial. EnCore samples should be extruded into methanol upon receipt. Record scintillation vial lot number, the sample weight, the methanol lot number, the preparation date and the analyst who prepared it in the 5035 preparation batch in TALS. Sample weights are calculated in the laboratory by adding the tare weight of the scintillation vial to the "Tare Weight" column and then entering the weight of the scintillation vial plus sample into the "Vial & Sample" column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples were preserved in MeOH at another TALS lab the calculated initial sample weight can be manually entered into the "Initial Amount" column of the preparation batch sheet for the corresponding sample container ID.

11.6.1.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are also extracted. To prepare the method blank (MB); add 10-mL of methanol to 10g Ottawa sand. To prepare the blank spike (LCS)/blank spike duplicate (LCSD), add 160-uL of the 8260 - working solution and 10-mL of methanol to 10g muffled Ottawa sand. (NOTE: The same metal spatulas to weigh soil samples must be used for measuring out the Ottawa sand).

11.6.1.3 If sufficient sample is available, one matrix spike (MS)/matrix spike duplicate (MSD) pair is extracted per extraction batch of up to 20 samples. If the sample set being extracted consists of products, waste oils, or other sample matrixes which based on analyst experience and discretion would not yield acceptable spike results due to matrix effects, a matrix duplicate (MD) and/or MD/MS may be prepared in lieu of and MS/MSD. To prepare the matrix spike (MS)/matrix spike duplicate (MSD), add 160 uL of the 8260 working solution and 10 mL of methanol to 10 g pre-weighed soil samples.

11.6.1.4 Add 5 mL (for EnCore™ or TerraCore™) or 10mL (in house soil extraction) of methanol to all vials immediately after recording sample weight.

11.6.1.5 Vortex the samples for the extraction batch for approximately 10 to 30 seconds to break up any large clumps in the extraction vials. This is especially important for extruded samples as they may be compacted in the EnCore™ or TerraCore™ sampling device and come out as a pellet. If after 30 seconds a pellet still remains, vortex for an additional 30 seconds.

If pellet still remains, further vortex mixing is not recommended, proceed to next step. It should be noted that the MB and LCS must be vortex mixed the same amount of time as the longest associated sample.

11.6.1.6 After all samples have been vortex mixed, place all samples for the extraction batch into shaker box and set timer for *10* minutes. It is recommended that the caps of all vials are checked and tightened before placing in shaker box to prevent leaking. If samples are present which still contain pelletized sample after vortex mixing in step 11.6.1.5, set the timer for 10 to 15 minutes. It is not recommended to shake samples for more than 15 minutes. If a sample still contains pelletized sample after shaking for 15 minutes, vortex the sample for an additional 15 seconds and shake entire batch for additional 5 minutes. Any pelletized sample remaining after second shaking is noted in and NCM in the extraction batch.

11.6.1.7 After samples are shaken, place samples four at a time into centrifuge with inserts for scintillation vials. Spin samples for a sufficient time to create a transparent but not necessarily uncolored layer of methanol extract above the extracted material. The time will vary depending on the nature and particle size of the extracted material. Three to five minutes at 50% speed is usually sufficient. Again it should be noted that the MB and LCS must be centrifuged at the same rate and for the same time as the longest centrifuged associated sample.

11.6.1.8 *Medium*-level solid extracts are stored in the scintillation vial used for extraction and are stored at 0-6^{o}C. The extracts are removed from cold storage and are allowed to return to ambient temperature prior to analysis.

11.6.2 Sample extraction for *Medium*-Level Analysis, field preserved.

11.6.2.1 Each containers tare weight is recorded in the TALS preparation batch. Most containers will contain a bar code with the tare weight information that can be scanned for automatic entry into the tare weight entry field in the preparation batch. Sample weights are calculated in the laboratory by adding the received weight of the sample jar to the “Vial & Sample” column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples received are in 4 oz jars with 25 mls of methanol (AK samples) the calculated initial weight of the sample must be adjusted to correct for the weight of the methanol which is not included in the container tare weight. TALS will perform this calculation, however the analyst must enter a “1” into the “Tare Incl MeOH” column of the preparation batch sheet for the corresponding sample container ID. The nominal amount of initial soil should be 10g for VOA vials with 10mL of methanol, or 25g with 25mL of methanol. When the calculated initial weight of the soil deviates by more than 20% of the expected value, high or low, an NCM should be written detailing that the 1:1 ratio of soil:methanol is significantly exceeded. Acceptable weights are 8g-12g for a 10g:10mL sample, and 20g-30g for 25g:25mL sample.

11.6.2.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are prepared by the laboratory prior to sample analysis. A MB and an LCS sample consists of 10g of muffled Ottowa Sand added to a scintillation vial followed by 10-mL of methanol.

11.6.2.3 Add the correct amount of matrix spiking solutions to all LCS samples. An aliquot of 160 uL of the 8260 working solution is added to 10mL extracts. The addition of the spike solutions introduces a slight error, which can be

neglected in the calculations. The listed volumes are halved (80 uL) for 5-mL extracts.

11.6.2.4 The MB, LCS and any received samples which appear to contain "clumps" of sample which could be broken up with agitation are vortex mixed for up to 1 minute. If no samples require agitation, only the MB and LCS are vortex mixed for approximately 15 seconds to ensure mixing of the sand and added solutions.

11.6.3 Sample preparation for Low-Level Analysis EnCore™.

11.6.3.1 VOA samples received in EnCores should be extruded into VOA vials with stir bars at time of receipt prior to storage in the freezer. Prior to analysis, low level soil samples collected or extruded to VOA vials with or without water are removed from the freezer and allowed to thaw. Samples should not be allowed to remain at room temperature for more than 30 minutes prior to extrusion into a VOA vial or analysis.

11.6.3.2 The weight of the samples is accurately recorded by either direct connection from the analytical balance to the batch record (preferred method) or by typing the balance reading into the batch record.

11.6.3.3 The extruded sample or the sample received in a VOA vial without water has 5 mls of reagent water added to it.

11.7 Sample Analysis

11.8 Preliminary Evaluation

11.8.1 Sample screening

Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories. Refer to section 11.15 for Dilutions.

11.9 Sample Analysis Procedure

11.9.1 All analysis conditions for samples must be the same as for the continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.9.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD (if sufficient sample volume allows), an LCS, and a method blank.

11.9.2.1 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.9.2.2 It is not necessary to reanalyze batch QC (except for the method blank) with reanalysis of samples. However, any re-runs must be as part of a valid batch.

11.10 Water Samples

11.10.1 All samples and standard solutions must be at ambient temperature before analysis.

11.10.2 For low-level analysis water samples are sub sampled by the autosampler at the appropriate volume (10 mL).

11.10.3 For standard level analysis 5 mLs are sub sampled by the autosampler.

11.10.4 For both low-level and standard level analysis internal standards and surrogates are added by the autosampler. Refer to Tables 5 and 6.

11.10.5 MS/MSD samples are prepared by injecting the 8260 working solution through the VOA vial septa using a bevel tipped syringe or directly into the MS or MSD VOA vial.

11.10.6 Purge the sample at ambient temperature with a trap temperature of 25^{O}C.

11.10.7 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.10.8 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is *0-1* minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260^{O}C, and bake temperature is 220 to 270^{O}C.

11.10.9 Immediately after analysis or immediately after opening the sealed VOA vial and obtaining the necessary aliquots for dilutions, the analyst must check the pH of aqueous samples with narrow range pH paper to ensure the pH is < 2. Record the pH of aqueous samples and the lot number of the pH paper used on the analytical batch sheets along with any dilution factors used. In those cases where the pH is > 2, initiate a non-conformance report and qualify the data, noting if the sample(s) was analyzed outside of the shortened seven-day hold time or zero-day hold time for aromatics.

11.11 TCLP Leach Samples

11.11.1 Follow the instructions for standard level water samples using a 100 times dilution for the method blank, laboratory control sample, and all samples. An MS/MSD pair must be prepared per batch by the method described in section 11.10.6.

11.12 Methanol Extract samples

11.12.1 Fill a VOA vial with reagent water, and remove 900 uL of water using a volumetric pipette.

11.12.2 Add 1075 uL of methanolic extract to the vial and immediately cap the VOA vial invert the vial to ensure that no air bubble larger than 4 mm is present. If there is an air bubble and it is greater than 4 mm, re-prepare sample.

11.12.3 The final volume of reagent water and methanolic extract used is entered into Chrome which then is uploaded into the analytical batch in TALS (43 mls).

11.12.4 As with water samples, internal standards and surrogates are added by the autosampler. Refer to Tables 5 and 6. TFT may be also be added to the field methanol (Alaska samples), thus the additional amount of TFT should be taken into account in the prep batch for AK samples.

11.12.5 Load the sample in the autosampler and proceed to analyze.

11.12.6 MS/MSD samples for in house extracts are prepared at time of extraction and are prepared for analysis as above. For field preserved samples, an in house post spike of the prepared sample is necessary, and is prepared by injecting the 8260 working solution through the VOA vial septa using a bevel tipped syringe.

11.12.7 Dilutions of methanolic extracts are made by adding proportional amounts of methanol extract to a new VOA vial. Samples of greater than 5x do not require the removal of water from the VOA vial prior to addition of methanolic extract.

11.12.8 Purge the sample at ambient temperature with a trap temperature of 25^{O}C.

11.12.9 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.12.10 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is *0-1* minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260^{O}C, and bake temperature is 220 to 270^{O}C.

11.13 Low-Level Solids Analysis using Discrete Autosamplers

11.13.1 Sample collection and initial preparation for low level soil samples has been discussed in section 8.9 and 11.6.

11.13.2 As with water samples, internal standards and surrogates is added by the autosampler. Refer to Tables 5 and 6.

11.13.3 MS/MSD samples for low level soil samples prepared by injecting the 8260 Working Solution through the VOA vial septa using a bevel tipped syringe. Direct purge soil analyses are spiked during the preparation of the extraction batch through the septa after capping (as applicable).

11.13.4 When it is feasible to perform dilutions of low level soils, a smaller amount of sample is weighed and analyzed. When dilutions of low level soil samples are not possible, any analyte that exceeds the calibration range must be E flagged on the appropriate reporting form, NCM filled out, and must be noted in the case narrative. In addition, if sufficient volume was provided, a methanolic extract must be prepared and analyzed.

11.13.5 Purge the sample at ambient temperature with a trap temperature of 25^{O}C.

11.13.6 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.13.7 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 1 minute or 2 minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260^{O}C, and bake temperature is 220 to 270^{O}C.

11.14 Initial Review and Corrective Actions

11.14.1 Retention Times

For 8260B if the retention time for any internal standard in the continuing calibration changes by more than 30 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

For DoD projects of 8260C if the retention time for any internal standard in the continuing calibration changes by more than 10 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.14.2 Internal Standard Response

If the internal standard response in the daily continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

For non-DOD samples, the internal standard response in a sample is compared to the internal standard response in the daily continuing calibration. For DOD samples, the internal standard response in a sample is compared to the internal standard response of the mid point standard in the initial calibration (not the daily CCV). For this reason, separate analytical sequences need to be employed for DOD samples. Responses from

50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary, though may be required to prove matrix effect. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected. If the ISTD response falls below 50% and the sample has no target analyte detections above the RL (1/2 the RL for DoD projects), the data may be qualified and reported. This should be done in consultation with the PM and client, an NCM must be written.

11.14.3 Surrogate Standard Recoveries

The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect (this may be demonstrated by a MS/MSD analysis as well). Reanalysis at a dilution should be considered if appropriate. If a diluted analysis is necessary and surrogate recoveries are in control or less affected, this is sufficient to demonstrate matrix interference.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

11.15 Dilutions

Dilutions *for waters are made directly into a 40mL VOA vial and* should be *prepared* just prior to the GC/MS analysis of the sample. *For example, a 10X dilution is made by filling a VOA vial to slightly overfull and then removing 4.3 mL of reagent water from the vial. 4.3 mL of sample is then transferred to the vial to bring it back to full and it is immediately capped for analysis. In the worklist this is recorded at a 10.0 dilutiuon.*

Soil dilutions are prepared by adding a smaller aliquot than the standard 1.075mL or by serial dilution for aliquots smaller than 20uL or viscous samples. In the prep batch this is recorded as the amount added to 43 mL. The worklist is left as a 1.0 dilution.

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample/extract is prepared and analyzed. A dilution should be prepared to ensure that the majority of compounds being diluted for fall in the middle to upper part of the calibration curve (i.e. from 40-90% of their respective calibration range). All reported dilutions must be within the calibration range of the respective analytes and should be compared to other dilutions to ensure that the diluted data “makes sense” as a check for possible dilution errors. If this cannot be accomplished with a single dilution, multiple sample dilutions *may be* necessary. Dilution levels should be considered carefully and it is recommended that a “complicated” sample (one which has more than three compounds which require dilution) is discussed with another analyst or area supervisor as necessary in order to minimize the number of dilutions required. Samples may be screened to determine the appropriate dilution for the initial run or historical site data may be used to determine initial dilutions.

11.15.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment and reasonable client requirements and requests.

11.15.2 Reporting Dilutions

The most concentrated dilution will be reported as the base dilution. Other dilution levels will report only the required diluted compounds and all other compounds and surrogates in the dilution will be set to acceptable in the LIMS system and not reported. Other reporting techniques may be required by specific project requirements or client request and will be transferred to the laboratory by the PM using the mechanisms previously discussed. See Section 9.1.3.

11.15.3 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP TA-WC-0125 for determination of percent moisture.

11.16 Instrument Maintenance

11.16.1 Agilent 5973 Inert, 5973 Network, 5975, 5975B, and 6890N

11.16.1.1 All circuit boards and peripheral attachments are dusted and vacuumed of debris and all plumbing and electrical connections inspected and adjusted; any replacement of worn parts is also done at this time.

11.16.1.2 The injection port is cleaned of debris by removing the injector and column nut and forcing clean methanol through the top of the injector into a waste container at the bottom. The inlet liner and gold seal are replaced as needed (e.g. visibly dirty or discolored, active site issues, etc.) The ion source is disassembled, cleaned, and reassembled with new filaments and insulators, if needed.

11.16.1.3 If low level soils cause recoveries for the internal standard to report low or purge flow to decrease in any consequent CCV's, the pencil filters or injector needle could be clogged. Methanol rinses of these parts may solve the issue, or replacing the part.

11.16.2 Column installation is performed when the following conditions are encountered;

- Heavy column bleed that cannot be eliminated by thermal conditioning.
- Loss of early eluting peaks due to column cutting.
- Inability to chromatographically resolve method performance compound peaks.
- Distortion of peak shapes i.e.; broadening, ghost peaks, split peaks that can't be resolved by injection port maintenance or flow control.

11.16.2.1 Turn the GC oven off and let the system cool to room temperature. Remove the column nut, liner, septum, and press tight inlet connector. Dispose of old column appropriately.

11.16.2.2 Cut approximately six inches off of the end of new columns. Install new column using appropriate sized ferrules and nuts.

11.16.2.3 Turn the GC on and set the injector temperature to 230 ℃, oven to the manufacturers recommended isotherm temperature or 10℃ below the manufacturers max temperature if an isotherm is not provided and condition for five minutes.

11.16.2.4 Perform an air water check on the system. When the air water spectrum shows acceptable levels, proceed with the mass calibration procedure. For additional information of column replacement see the manufacturer's operator's manual.

11.16.3 *OI Analytical 4560 and 4660* Purge and Trap Concentrator Units

11.16.3.1 A new *OI #10* trap is installed and conditioned by *baking at 210 degrees C for at least 30 minutes.*.An initial conditioning of overnight is recommended as time allows.

11.16.3.2 Sample lines, internal valves, sparge cells, and sparge cell mounts and fittings are rinsed with purge and trap grade methanol or replaced as necessary.

11.16.3.3 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.16.3.4 The purge gas flow rate (40 mL/min ± 5 mL/min) should be measured at the vent and recorded in the maintenance logbook.

11.16.4 Archon 2000 or equivalent type auto sampler.

11.16.4.1 Remove debris and perform a calibration per manufacturer's instructions.

11.16.4.2 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.16.4.3 The guide rails are wiped down with a Kim wipe and *n-propanol* to remove grease buildup and debris. *Arms, rods, and rails should be removed and all parts cleaned at least once per year, especially bearings.*

11.16.4.4 The syringe is cleaned per manufacturer's instructions to remove debris.

11.16.4.5 The *rinse* reservoir is refilled *with DI (standard level methods) or purge water (low level methods)* prior to analysis.

11.16.4.6 The Internal Standard/Surrogate Standard vial is refilled as needed and primed after filling to ensure the instrument is drawing volume.

11.16.5 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the source and replacing the multiplier. In addition, a new initial calibration may be necessary if a large amount of routine maintenance occurs at once. This will be determined by evaluating the system using a CCV.

11.16.6 Maintenance Logbook

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, tune, etc as may be the case).

If columns were replaced during maintenance procedures, the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook.

11.17 Troubleshooting

11.17.1 QUESTIONS TO ASK YOURSELF

11.17.1.1 MECHANICAL ISSUE?

- Has the problem just started, or has it been getting worse with time?
- Has anything recently been done to the instrument that may have caused the problem to start?
- Have there been any nasty samples run on the instrument recently?

11.17.1.2 ANALYTE ISSUE?

- Are there any other compounds out or trending out?
- Did the CCV and/or LCS show similar trends?
- Is this a one time event? Or did you see similar signs yesterday?
- Is it failing on other mass specs?
- Did anything change from the last time it passed?
- Have there been any nasty samples run on the instrument recently?

11.17.1.3 INTERNAL/SURROGATE ISSUE?

- Have they been stable up until now?
- Is the gas on and are the standards fresh?
- Are the glass vials correctly positioned and tight?
- Do they look like they are trending in any direction or just fluctuating?
- Is the tune stable? Are the ratios close to the ICAL tune?

11.17.2 BEST PRACTICE

11.17.2.1 If you can – replace the most likely piece in the system, label the old one with which instrument it's from, the date out and possibly the problem you are having e.g. R1, 071010 Bromoform. Test if the problem is fixed. If not, move onto the next thing and replace until the problem is fixed. Then work backward and reinstall the old pieces which are still OK. If you no longer need a part that is still good, label it as such. If a piece is bad and not even good for testing with throw it out and tell your department manager what piece and part number.

11.17.3 TYPICAL FIRST THINGS TO LOOK AT AND TRY

11.17.3.1 CHECK YOUR FLOWS (For soil mode, purge on archon)

- Set the Archon to purge for 111mins instead of 11mins then set a vial to purge and "pause" the concentrator. This will give ample time to troubleshoot.
- Use the flow meter to check the flow out the purge vent using a small clear tube as an adapter – you should have about 40mls.
- Take the transfer line off at the entrance to the concentrator and measure the flow – you should also have about 40mls.
- Generally this would suggest you don't have a blockage or a leak, but check the following first
- Find a clear tube that has been blocked at one end, place this on the purge vent while the vial is purging and establish the following:
- Did the bubbles stop straight away <2mins?
- Yes – the leak is in the Archon
- Have the bubbles stopped over a 5 – 7 min period?
    - Yes – Most probably not a leak but could be a blockage (which includes the tube you put on the vent)
    - No – There is a leak somewhere, most likely the concentrator.

- If there is no soil valve attached then remove the transfer line inlet (to concentrator) fitting and replace with a capped fitting, Run the concentrator full leak check.
- If all of the above passes then you do not have a problem with the flows/blockage/leak.
- Remember to set Archon back to 11mins purge and bake off trap before running again.

11.17.3.2 IF YOU HAVE A LEAK

- In the Archon – purge a vial and leak check around the top of the needle, the transfer line and the pencil filter in the back of the Archon.
- In the concentrator – with a vial purging leak check around all the fittings starting with the entry from the transfer line, the 4-port valve, the 6-port valve ad around the water management and trap.
- You can also check the flows coming out of the 4-port, leaving the 6-port, there is an adapter which can be added instead of the trap to measure through the water management, from the trap. If all of these measures around 40mls and still you have less than 37 coming out the purge vent it could be leaking across the valves in the side of the concentrator.
- REMEMBER – any Archon that has been changed to Nitrogen will not be able to be checked with the leak detector.

11.17.3.3 COMMON PLACE TO LOOK IF IT'S A ANALYTE PROBLEM

- **1,1,2,2-Trichloroethane RRF** – Could be any of the lines as often an active site. Start with transfer line, lines in concentrator, water management and trap, soil valve if installed.
- **Bromoform** – Flow is not 40mls, water management, transfer line, small lines in concentrator. This can sometime be a tune issue as well if it is somewhat different from normal.
- **Chloromethane carryover** – Often the trap, replace the trap with the wire at the bottom and condition at 210^{o}C for 30min.
- **Napthalene carryover/low response** – Often water management, change this out and condition with 5 or 6 varying concentrations of an old main or 524 std. e.g. 20ppb, 80ppb, 10ppb, 50ppb etc. Could also be transfer line.
- **2-CEVE** – could be anywhere. Start with transfer line and work way through system, small lines in concentrator, purge needle as last resort. Could also be water management, soil valve if installed.
- **MTBE** – often an active site could be transfer line or trap. Sometimes the instrument just needs recalibrating.
- **Vinyl Chloride** – Usually the first compound to fail low in the standard mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **1,1-Dichloroethene** – Usually the first compound to fail low in the Main mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **Chloroform** – usually your tune is differing from the ICAL
- **Contamination** – look back at previous runs, if you have high hits for this compound then run 20 – 30 blanks to clean it up. If you still have high concentrations then the sample possibly blew back into the instrument so could be purge valves or the pencil filter in the back of the Archon.
- **Ethylbenzene** – usually your tune is differing from the ICAL.

11.17.3.4 PEAK SHAPE DIFFERENT

- If you have peak tailing on the front end compounds such as Vinyl Chloride, Chloromethane etc then it is often a result of a worn trap.

11.17.3.5 SURROGATES FAILING

- If you have 1,2-DCA failing and all the other surrogates are OK, then the likely cause is the tune. Often the ratios will have moved. Follow the surrogates, if low, then drop ratios etc
- If you have all the surrogates out and it seems to be consistently that way, it could be the following,
    - Is the gas turned on?
    - Is the surrogate vial positioned properly on the archon
    - Your concentration levels in the ICAL maybe incorrect if you have just ICALed.
    - The valve has perhaps worn a little and the nominal value in target needs to be updated, but this can only happen after an ICAL.
    - If the surrogates need to be weighted linear or linear in an ICAL and you see them trending this way for a while its possible the column has gone bad or you may have a standard valve leak.
    - You can check to see if it's the archon causing the problem by manually injecting IS/SS and turn off the IS and SS on the archon
- If two surrogates are high and two are low, sometimes this is because your Archon surrogate is not matching with the surrogate you ICAL'd with. Try swapping out the Archon surrogate and if this does not fix it, re-ICAL the Supp with the new surrogate.

11.17.3.6 INTERNALS FAILING, TRENDING OR BOUNCING

- How fresh is your internals? If they are more than say a month old, they may have gone bad. Is the vial on properly and done up tight (not too tight).
- Is the gas turned on?
- How long has the instrument been using the current filament? More than 4weeks then you may want to change to the other or change the filaments if they are both spent.
- If the internals bouncing seems to correlate with the tune then most likely you want to change filament or clean the source.
- If the internals fall to a value and sit there or raise to a value and sit there you may want to consider ICAL it at that tune setting, instruments have a kind of sweet spot.
- Internals falling off maybe a water buildup and the parameters need looking at on the concentrator. Have a look at other instruments to see how they are set up and try one or two different settings to establish if this helps. Always keep a note of where they were at and what you have changed them too. Best place to record this is on your sequence and in the white folder under daily changes.
- Split ratios can also effect internals, try increasing the ratio to more like 45:1 if not already at this level.

11.17.3.7 CONCENTRATOR STOPS

- Turn it off and on again, it is common for the concentrator to lose the plot every so often.
- Shows a temperature error – check to make sure the trap is covered and the main cover is down properly, any heat escape will cause an error.

11.17.3.8 SEQUENCE STOPS

- You have started on a file that already exists and you have not checked the overwrite file box.

- Communication problem with the GC and MS. Turn off the MS and GC and restart the computer. Restart the MS and GC.

11.17.3.9 ARCHON STOPS

- Standard home errors – syringe already home – use the maintenance menu on the archon to step you through cleaning the syringe. Before putting back together smear the inside of the syringe with 'Nose grease'.
- Standard home errors -

12.0 Calculations / Data Reduction

12.1 Qualitative Identification for Full Scan Analysis

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1.1 The sample component retention time must compare to within ± 0.06 RRT units of the retention time of the standard component. For reference, the standard must be run within the same twelve hour tune as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.1.3 All data are subject to two levels of technical review, as described in SOP TA-QA-0635.

12.2 Tentatively Identified Compounds (TICs)

12.2.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:

12.2.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification. If the TIC requested is part of the list of target analytes, the TIC will not appear under the list of tentatively identified compounds.

12.2.1.7 Once tentative identifications are assigned, these results are uploaded into LIMS with the other data and the TICs are automatically reported to the client from the LIMS.

12.3 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

12.4 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

12.5 Response Factor (RF)

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng.
C_x = Concentration of the compound being measured, ng.

12.6 Standard deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of Xi.

12.7 Percent relative standard deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

12.8 Percent drift between the initial calibration and the continuing calibration:

$$\%Drift = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100\%$$

Where:

$C_{expected}$ = Known concentration in standard.
C_{found} = Measured concentration using selected quantitation method.

12.9 Concentration = mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

Note: For all methanolic samples with a % Moisture of greater than 10%, it is necessary to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect. This is done by the following equation:

Corrected FV = ((g of samples * % moisture/100) + ml of MeOH) * 40

In these situations, an "other observation" NCM must be generated and the correction and above formula must be noted in the case narrative.

12.10 Calculation of Results for Methanol Extracts

$$\text{Sample Conc (ug/Kg)} = [\text{On Column (ug/L)}] \times \frac{\text{Extraction Final Volume (mL)} \times \text{VOA Vial Volume (mL)} \times \text{1L (CF)} \times \text{1000g (CF)}}{\text{Amount of Soil Sample (g)} \quad \text{Amt of MeOH Extract (mL)} \quad \text{1000mL} \quad \text{1Kg}}$$

VOA vial volume is 43 ml and when the extract doesn't require a dilution, 1.075 mL of the methanol extract is used. So, the equation becomes:

$$\text{Sample Conc (ug/Kg)} = [\text{On Column (ug/L)}] \times \frac{\text{Extraction Final Volume (mL)}^{1} \times \text{43 (mL)} \times \text{1L (CF)} \times \text{1000g (CF)}}{\text{Amount of Soil Sample (g)}^{2} \quad \text{1.075 (mL)} \quad \text{1000mL} \quad \text{1Kg}}$$

[1]Extract Final Volume, miscible solvent corrected (mL) = ((g of samples * % moisture/100) + ml of MeOH) * 40
(used when % Moisture of the soil sample is greater than 10%).
[2]Amount of Soil, dry-weight corrected (g) = sample mass (g) * (100 - % moisture/100)

12.11 Upon completion of the analytical sequence:

12.11.1 Review chromatograms online and determine whether manual data manipulations are necessary.

12.11.2 Manual Integrations

All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

12.11.3 Manual integrations may be processed using Chrom, which stores the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

12.11.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

12.11.5 Confirm that run logs have printed on them the instrument ID, the analyst and the method used. If this is not printed on the run logs, this must be entered by hand prior to completing the package.

12.12 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 field samples.

12.12.1 Perform a level 1 data review and document the review on the data review checklist (GCMS Data Review Checklist).

12.12.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

12.13 Method Performance

12.14 Method Detection Limit Study (MDL)/Detection Limit

The method detection limit (MDL) or detection limit (DL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL/DL is determined according to the laboratory's MDL/DL procedure (see SOP TA-QA-0602). MDL/DLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL/DL studies for analyses performed; these are verified at least quarterly unless method requirements require a greater frequency.

For instruments that run samples which fall under LaMP regulations, a yearly MDL Study must be performed and MDLV starting at 1X the MDL.

12.15 Limit of Detection

The limit of detection (LOD) is determined for each analyte and matrix by spiking a quality system matrix at approximately two to four times the detection limit. This spike concentration establishes the LOD. The LOD is verified quarterly for each method and matrix on each instrument that analyzes said method/matrix. Refer to the laboratory's LOD procedure (see SOP TA-QA-0602)

12.16 Limit of Quantitation

The limit of Quantitation (LOQ) is verified quarterly for each method and matrix on each instrument that analyzed said method/matrix. Refer to the laboratory's LOQ procedure (see SOP TA-QA-0618)

12.17 Demonstration of Capabilities

Analyst initial Demonstrations of Capability (DOC) are performed after completing a read and understand memo for the SOP and before any client samples are analyzed. DOCs are updated annually (continuing DOC). See SOP TA-QA-0617 for details.

12.18 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Waste Disposal SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 VOA vials containing acidic water; VOA vials containing extracted acidic water and small amounts of methanol are collected in large plastic satellite waste bins marked "Hazardous Waste." At or before the waste reaches 55 gallons, the contents are transferred to the waste warehouse where the vials are bulked into a 55 gallon waste barrel and sent out for incineration.

14.1.2 VOA vials containing extracted soil samples, which will contain small amounts of methanol and possibly sodium bisulfate. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days have passed these sample extracts are transported to the waste room were they are bulked into the flammable liquid loose pack barrel and sent out for incineration.

14.1.3 Expired Standards. Expired standards are collected in satellite containers marked "Hazardous Waste." At or before the containers reach 55 gallons the containers are taken to the waste warehouse where they are bulked into an expired standards lab pack and sent out for incineration.

15.0 References / Cross-References

15.1 Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, August, 2006, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Fourth Edition, EPA Office of Solid Waste.

15.3 Method 5035A, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July, 2002, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.4 40CFR, Part 136, Appendix A (Method 624).

15.5 Department of Defense (DoD), Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009), DoD Quality Systems Manual Version 5.1 DOE Quality Systems for Analytical Services Version 3.1, 2017.

- **Method Modifications:**

Item	Method	Modification
1	8260B	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
2	8260C	Storage conditions for standards are listed as <6C or as recommended by manufacturer. TestAmerica Seattle follows 8260B guidelines to store standards at -10C or as recommended by manufacturer.
3	8260C	The minimum RF values for Chloroethane, Acetone, 2-Butanone, 4-Methyl-2-pentanone and 2-Hexanone are based on a lab study and not as listed in the method.
4	624	See Appendix A
5	5035A	The aliquot of methanol extract taken for analysis is 125 uL rather than the 100 uL specified in Table 1 of the method.

- **Tables and Appendices**

Table 1 TestAmerica Primary *Analyte* List for 8260B and 8260C
Table A-1 Method 624 Analytes *5*-mL Purge
Table 2 8260B and 8260C Additional Analyte List
Table 3 Appendix IX List
Table 4 Typical Calibration Levels
Table 5 Internal Standards
Table 6 Surrogate Standards
Table 7 Short List LCS and Matrix Spike Standard
Table 7a Full List LCS and Matrix Spike Standard
Table 8 BFB Key Ion Abundance Criteria
Table 9 8260B SPCC Compounds and Minimum Response Factors
Table 10 8260B CCC Compounds
Table 11 Poorly Performing Compounds
Table 12 CCV % Drift Criteria for Non-DOD Projects
Table 13 Summary of QC Requirements
Table 14 8260C Minimum Relative Response Factor Criteria
Appendix A Modifications for Method 624
Appendix B Process Flow for Seattle VOA

- **Changes from last revision**

- Revision 28, dated 27 June 2017
  - Changed references to ‘high’ level soils to ‘medium’ level
  - Updated Approvals
  - Removed references to CA-LUFT, AK101, and NWTPH-GX by MS
  - Updated Calibration Ranges
  - Updated sand preparation method
  - Updated reagents
  - Added requirement for 1:1 ration for methanol soils
  - Clarified acceptable drift ranges of poor performing compounds
  - Updated section 11.15 Dilutions
  - Updated section 11.16 Instrument Maintenance

- Revision 27, dated 19 July 2016
    - Added requirement in section 11.4.16 to remove analyte calibrations which do not pass % drift criteria in ical to not calibrated.
    - Updated section 1.41 Calibration working ranges.
    - Removed from section 3.9 use of second source standard for LCS, MS, and MSD.
    - Updated section 6.1 to current column used.
    - Removed 10 mL glass syringe from section 6.3.
    - Updated section 8.9.3.2 to 5 mL DI water volume for stir bar vials.
    - Updated section 11.4.16 to current ICV concentrations.
    - Updated Approvals.

- Revision 26, dated 29 March 2016
    - Updated sections 8.9.1, 11.6.1.1 and 11.6.3.1 to clarify the requirement to extrude Encores upon receipt.
    - Updated section 11.11 to new standard level water procedures.
    - Updated section 11.13 to new methanol, medium-level soil, procedures.
    - Updated section 11.14 to new low-level soil procedures.
    - Updated calculation in section 12.10 to current procudures.

- Revision 25, dated 5 March 2015
    - Section 9.2 of Appendix B, added NWTPH-Gx batch criteria
    - Section 9.5.2 and 9.5.3 of Appendix B, added surrogate recovery criteria for AK101 and NWTPH-Gx
    - Section 10.1.4 and 11.9.4 of Appendix B, added CCV acceptance criteria per method.
    - Section 11.4.1 and 11.4.3 of Appendix B, added NWTPH-Gx criteria for the ICAL
    - Section 11.7.4 and 11.7.5 of Appendix B, added ICV acceptance criteria per method.
    - Section 15.5 of main body SOP and 15.1.3 of Appendix B, updated reference for the DoD QSM.

- Revision 24, dated 5 January 2015
    - Section 1.2, included gas by MS to list of methods
    - Section 5.1.1 updated heat concerns to include other instrument parts
    - Section 6.1 updated sample purger section
    - Section 7.3.3 expounded on CCC failures
    - Section 8.2.1 added analytes
    - Section 8.2.2 deleted, 0 day holding time is no longer applicable
    - Section 8.5 expounded on acceptable criteria
    - Section 9.5 removed LCS criteria to conform to DoD QSM 5.0 standards
    - Section 11.4.8 some RF values changed due to in-house investigation
    - Section 11.10.3 updated 5030 preparation method to correct audit finding
    - 11.10.10 updated recorded values of dilutions to correct audit finding
    - Added section 11.16.1.3
    - Section 12.2.1.6 updated TIC evaluation
    - Added Appendix B: Gas Analysis by GC/MS based on SW-846, CALUFT, AK101, and Northwest Methods
    - Table 14 updated RF values based on in lab study

- Revision 23, dated 5 January 2014
    - Section 2.3, updated purge gas.
    - Section 6.2, added computer hardware
    - Section 8.3, updated to clearly define different soil sample techniques
    - Section 8.5, updated container
    - Secton 8.6.2, updated container and volume of MeOH
    - Section 8.8, added section for unpreserved soils
    - Section 8.9.2, added VOA vials for low-level soils.
    - Section 9.3, added clarification for DoD and LaMP program requirements

- Sections 9.4 and 9.5, added program requirements
- Section 11.3, updated BFB criteria for mass 174
- Section 11.4 updated to current practice
- Section 11.4.4, added reference to corporate SOP on calibrations
- Sections 11.4.8, 11.4.9, 11.4.11 and 11.4.12, added the criteria for 8260C
- Section 11.4.15, added clarification for DoD and LaMP program requirements
- Sections 11.5.1 and 11.5.2, added DoD QSM 5.0 requirement for closing CCV
- Section 11.5.5, added the criteria for 8260C
- Section 11.5.7, added DoD QSM 5.0 requirement for closing CCV
- Sections 11.5.8 and 11.5.9, added clarification for DoD and LaMP program requirements
- Sections 11.6.1.1, 11.6.2.1, 11.6.3.1 and 11.6.3.3, added more detail for the procedure.
- Sections 11.10, 11.12 and 11.13, updated to new procedures
- Section 11.14.1, added the criteria for 8260C
- Section 11.15, updated to new procedures
- Section 11.17, added section for troubleshooting
- Updated and added Tables and Attachments

- Revision 22, dated 13 March 2013
  - Added section 11.7 to describe the bulk soil preparation procedures and documentation.
  - Added section 15.1 Waste Streams Produced by the Method.

- Revision 21, dated 12 October 2012.
  - Added an elaborated calculation for methanolic extracts 12.10.1.

- Revision 20, dated 20 June 2011
  - Incorporated ROMDs 00019 and 00026 in sections 6.1 and 11.16.6.
  - Added “Note” in section 4.4 addressing cross-contamination from laboratory spiked samples.
  - Addressed shelf life of gas standards in section 7.3.2.
  - Add instructions to return standards to freezer when daily prep is completed, section 7.4
  - Added hold time NCM for unpreserved aromatic compounds in Section 8.2.
  - Added clarification in section 8.5.2 that TCLP samples require a MS/MSD.
  - Added hold time for unpreserved aromatic compounds in Section 8.13.
  - Added clarification on spike solutions in sections 9.5 and 9.6.
  - Incorporated ROMD 00025 in section 9.6.
  - Incorporated ROMD 00020 in section 11.2.2.
  - Added optimization objectives for the GC run programs in section 11.2.2.
  - Incorporated ROMD 00022 in section 11.5.12.
  - Incorporated ROMD 00033 in section 11.6.3
  - Incorporated ROMD 00024 in section 11.6.8.
  - Updated corrective actions for CCV failure in section 11.6.8.
  - Defined CCV % Drift Criteria for non-DOD projects in section 11.6.6 and Table 12
  - Added section 11.11 for TCLP sample preparation/analysis.
  - Added pH check procedures for water samples in section 11.10.9.
  - Revised IS acceptance criteria in section 11.14.2.
  - Expanded list of compounds in Table 7 for compliance with NELAC.

- Revision 19, dated 16 September 2010
  - Updated corrective actions for ICV and CCV failures, sections 11.5 and 11.6.
  - Left italicized text from revision 18 since this revision occurred just days after.

- Revision 18, dated 13 September 2010
  - Added more detail to tuning procedures, section 11.
  - Updated corrective actions for ICV and CCV failures, sections 11.5 and 11.6.
  - Added qualitative identification requirements for SIM analysis, section 12.2

- Revision 17, dated 16 April 2010
    - Removed Chlorobenzen-D5 from Table 5. Added Pentafluorobenzene and 1,4-Dichlorobenzene and updated quantitation ion.
    - Added Sections on 8260_SIM. Including MS parameters and recommended ICAL and spike ranges. Updates Scope section 1.2.1. Updated Calibration 11.4.2. Added section 11.2.3.1 SIM parameters
    - Added a range of spiking amounts and added 10mL purge spiking amounts to sections 11.9
    - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
    - Added clarifications about standard storage conditions, section 7.3.1
    - Added removal of expired standards Section 7.4.
    - Added instructions for the analysis of storage blanks, section 8.11
    - Added clarification about re-analyzing batch QC and trip blanks, section 9.7
    - Added criteria for additional QC, Section 9.8.
    - Added daily balance check to Section 6.2.
    - Added LaMP client surrogate criteria, Section 9.4.
    - Added requirement for running 624 and 8260 methods on separate sequences Section 10.1.
    - Added measurement of gas flow rate, Section11.14.3.4.
    - Added maintenance logbook documentation requirements, section 11.14.5
    - Added specifications (MDLs) and clarifications (DOCs), section 13
    - Updated appendix A to include purge and desorb requirements for method 624.
    - Integration for TestAmerica Seattle and TestAmerica Tacoma operations.

- Revision 16, dated 16 September 2009
    - Description of preparation requirements for reagent water have been updated in sections 3.3 and 7.1.2 to reflect current practice of by purging with an inert gas for a minimum of 1 hour prior to use rather than for a period of overnight.

- Revision 15, dated 22 July 2009
    - Method modifications section updated to identify volume of methanol extract used for analysis.
    - References section updated to include Method 5035A.
    - Nomenclature for spiking solutions has been updated.
    - Spiking volumes have been updated.
    - Calibration levels have been updated.
    - Corrected typographical errors.
    - Updated RLs in Tables 1, 2, 3, and A-1.
    - Added Table 12. Summary of QC Requirements

- Revision 14, dated 16 March 2009
    - Method modifications section updated to identify volume of methanol extract used for analysis.
    - References section updated to include Method 5035A.
    - Nomenclature for spiking solutions has been updated.
    - Spiking volumes have been updated.
    - Calibration levels have been updated.
    - Corrected typographical errors.
    - Updated RLs in Tables 1, 2, 3, and A-1.

- Revision 13, dated 22 March 2008
    - Integration for TestAmerica and STL operations.
    - This revision is a complete rewrite and an expansion of scope.
    - This SOP is the combination of SOPs 0312.12, 0327.5, and 0381.6.

Table 1

TestAmerica Primary Analyte List for 8260B and 8260C

Current reporting limits for all methods may be found in TALS through the following pathway: Global Method Data > Methods > Select Method on left-hand column > Limits View tab > Select Sample Matrix > Select Limit Type "RL"

Compound	CAS Number
Dichlorodifluoromethane	75-71-8
Chloromethane	74-87-3
Bromomethane	74-83-9
Vinyl chloride	75-01-4
Chloroethane	75-00-3
Trichlorofluoromethane	75-69-4
Acetone	67-64-1
Carbon disulfide	75-15-0
Methylene chloride	75-09-2
1,1-Dichloroethene	75-35-4
1,1-Dichloroethane	75-34-3
trans-1,2-Dichloroethene	156-60-5
Methyl tert-butyl ether (MTBE)	1634-04-4
cis-1,2-Dichloroethene	156-59-2
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
Dibromomethane	74-95-3
2-Butanone (MEK)	78-93-3
1,1,1-Trichloroethane	71-55-6
Carbon tetrachloride	56-23-5
Bromodichloromethane	75-27-4
1,2-Dichloropropane	78-87-5
cis-1,3-Dichloropropene	10061-01-5
Trichloroethene	79-01-6
Dibromochloromethane	124-48-1
1,2-Dibromoethane (EDB)	106-93-4
1,2,3-Trichloropropane	96-18-4
1,1,2-Trichloroethane	79-00-5
Benzene	71-43-2
trans-1,3-Dichloropropene	10061-02-6
Bromoform	75-25-2
4-Methyl-2-pentanone (MIBK)	108-10-1
2-Hexanone	591-78-6

Table 1

TestAmerica Primary Analyte List for 8260B and 8260C

Current reporting limits for all methods may be found in TALS through the following pathway: Global Method Data > Methods > Select Method on left-hand column > Limits View tab > Select Sample Matrix > Select Limit Type "RL"

Compound	CAS Number
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,1,2,2-Tetrachloroethane	79-34-5
Chlorobenzene	108-90-7
Ethylbenzene	100-41-4
Styrene	100-42-5
m- and p-Xylenes	136777-61-2
o-xylene	95-47-6
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
2,2-Dichloropropane	590-20-7
Bromochloromethane	74-97-5
1,1-Dichloropropene	563-58-6
1,3-Dichloropropane	142-28-9
Bromobenzene	108-86-1
n-Propylbenzene	103-65-1
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
1,3,5-Trimethylbenzene	108-67-8
tert-Butylbenzene	98-06-6
1,2,4-Trimethylbenzene	95-63-6
sec-butylbenzene	135-98-8
4-Isopropyltoluene	99-87-6
n-Butylbenzene	104-51-8
1,2,4-Trichlorobenzene	120-82-1
Naphthalene	91-20-3
Hexachlorobutadiene	87-68-3
1,2,3-Trichlorobenzene	87-61-6
1,1,1,2-Tetrachloroethane	630-20-6
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
Isopropylbenzene	98-82-8

Table A-1

Method 624 Analytes *5*-mL Purge

Analytes	*CAS #*
Acrolein	*107-02-8*
Acrylonitrile	*107-13-1*
Benzene	*71-43-2*
Bromodichloromethane	*75-27-4*
Bromoform	*75-25-2*
Bromomethane	*74-83-9*
Carbon tetrachloride	*56-23-5*
Chlorobenzene	*108-90-7*
Chloroethane	*75-00-3*
2-Chloroethyl vinyl ether	*110-75-8*
Chloroform	*67-66-3*
Chloromethane	*74-87-3*
Dibromochloromethane	*124-48-1*
1,2-Dichlorobenzene	*95-50-1*
1,3-Dichlorobenzene	*541-73-1*
1,4-Dichlorobenzene	*106-46-7*
1,1-Dichloroethane	*75-34-3*
1,2-Dichloroethane	*107-6-2*
1,1-Dichloroethene	*75-35-4*
trans-1,2-Dichloroethene	*156-60-5*
1,2-Dichloropropane	*78-87-5*
cis-1,3-Dichloropropene	*10061-01-5*
trans-1,3-Dichloropropene	*10061-02-6*
Ethylbenzene	*100-41-4*
Methylene chloride	*75-09-2*
1,1,2,2-Tetrachloroethane	*79-34-5*
Tetrachloroethene	*127-18-4*
Toluene	*108-88-*
1,1,1-Trichloroethane	*630-20-6*
1,1,2-Trichloroethane	*79-00-5*
Trichloroethene	*79-01-6*
Trichlorofluoromethane	*75-69-4*
Vinyl chloride	*75-01-4*

Table 2

8260B and 8260C Additional Analyte List

Compound	CAS Number
Acrolein	107-02-8
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Iodomethane	74-88-4
2-Chloroethyl vinyl ether[1]	110-75-8
Vinyl acetate	108-05-4
Methyl acetate	79-20-9
2-Methyl-2-propanol	75-65-0
tert-Butyl Ethyl Ether	637-92-3
trans-1,4-Dichloro-2-butene	110-57-6
Total Xylenes	1330-20-7
Acrylonitrile	107-13-1
Hexane	110-54-3
Tetrahydrofuran	109-99-9
Cyclohexane	110-82-7
tert-Amyl Methyl Ether	994-05-8
Methylcyclohexane	108-87-2
cis-1,4-Dichloro-2-butene	1476-11-5
Hexachloroethane	67-72-1
2-Ethyl-1-hexanol	104-76-7
1,3,5-Trichlorobenzene	108-70-3

1 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 3

Appendix IX Analyte List

Compound	CAS Number
Acetonitrile	75-05-8
Isopropyl ether	108-20-3
n-Butanol	71-36-3
Methacrylonitrile	126-98-7
Isobutanol	78-83-1
Ethyl ether	60-29-7
Ethyl Acetate	141-78-6

Table 4

Typical Calibration Levels

Standard level water, Low-level Soil and Methanol preserved soil purge	**Calibration Levels, µg/L**										
	0.2	**0.4**	**1.0**	**5.0**	**10**	**20**	**50**		**100**		**150**
Low level water purge	**Calibration Levels, µg/L**										
	0.02	**0.05**	**0.10**	**0.20**	**0.40**	**1.0**	**5.0**	**10.0**	**25.0**	**40.0**	**50.0**

Table 5

Internal Standards

Internal Standard	Standard Concentration (mg/L)	Quantitation Ion
1,4-Dichlorobenzene-d4	250	152
Chlorobenzene-d5	250	117
Fluorobenzene	250	96
1,4-Dioxane-d8	5000	96
Tert-butanol-d9	5000	65

Table 6

Surrogate Standards

Surrogate Compounds	Standard Concentration (mg/L)
1,2-Dichloroethane-d4	150
4-Bromofluorobenzene	150
Dibromofluoromethane	150
Toluene-d8	150
Trifluorotoluene	150

NOTES:

1) Recovery and precision limits for the surrogates are generated from historical data and are maintained by the QA department.

Table 7

Full List LCS and Matrix Spike Compounds

Compound	Standard Concentration (mg/L)
Acrolein	250
Vinyl acetate	250
2-Chloroethyl vinyl ether	250
1,4-Dioxane	1250
2-Butanone (MEK)	250
2-Ethyl-1-hexanol	1250
2-Hexanone	250
2-Methyl-2-propanol	250
4-Methyl-2-pentanone (MIBK)	250
Acetone	250
Acetonitrile	500
Acrylonitrile	250
Bromomethane	250
Chloroethane	50
Chloromethane	50
Cis-1,4-Dichloro-2-butene	50
Dichlorodifluoromethane	50
Ethyl acetate	250
Ethyl ether	250
Iodomethane	250
Isopropyl ether	50
Methacrylonitrile	250
Methyl acetate	250
Methyl tert-butyl ether	50
Tert-amyl methyl ether	50
Tert-butyl ethyl ether	50
Tetrahydrofuran	250
Trans-1,4-Dichloro-2-butene	250
Trichlorfluoromethane	50
Vinyl chloride	50

Compound	Standard Concentration (mg/L)
1,1,1,2-Tetrachloroethane	50
1,1,1-Trichloroethane	50
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloropropane	50
1,3,5-Trichlorobenzene	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Chlorotoluene	50
4-Chlorotoluene	50
4-Isopropyltoluene	50
Benzene	50
Bromobenzene	50
Bromoform	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chlorobromomethane	50
Chlorodibromomethane	50

Compound	Standard Concentration (mg/L)
Chloroform	50
Cis-1,2-Dichloroethene	50
Cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromomethane	50
Dichlorobromomethane	50
Ethylbenzene	50
Ethylene Dibromide	50
Hexachlorobutadiene	50
Hexachloroethane	50
Hexane	50
Isobutyl alcohol	5000
Isopropylbenzene	50
Methylcyclohexane	50
Methylene chloride	50
m- & p-Xylene	100
Naphthalene	50
n-Butanol	5000
n-Butylbenzene	50
n-Propylbenzene	50
o-Xylene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50

NOTES:

1) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.

Table 8

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	50 to 120 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 9

8260B SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

Table 10
8260B CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30	≤ 20
1,1-Dichloroethene	≤ 30	≤ 20
Chloroform	≤ 30	≤ 20
1,2-Dichloropropane	≤ 30	≤ 20
Toluene	≤ 30	≤ 20
Ethylbenzene	≤ 30	≤ 20

Table 11

Poorly Performing Compounds

Acetone	Isopropyl alcohol
Acetonitrile	Dichlorofluoromethane
Acrolein	**1,2-Dibromo-3-chloropropane (DBCP)**
Acrylonitrile	1,4-Dioxane
n-Butanol	Ethyl acetate
2-Butanone (MEK)	**2-Hexanone**
Bromomethane	Methacrylonitrile
Bromoform	Methyl acetate
Carbon disulfide	Methyl methacrylate
2-Chloroethyl vinyl ether	**4-Methyl-2-pentanone (MIBK)**
Chloroethane	**Naphthalene**
Chloromethane	2-Nitropropane
Dichlorodifluoromethane	Proprionitrile
cis-1,4-Dichloro-2-butene	Tetrahydrofuran
trans-1,4-Dichloro-2-butene	1,1,2-Trichloro-1,2,2-trifluoroethane
Ethanol	**1,2,3-Trichlorobenzene**
Ethyl methacrylate	**Trichlorofluoromethane**
Iodomethane	Vinyl acetate
Isobutyl alcohol	Tert butyl alcohol

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer." This is not a comprehensive list and is subject to change. Each DoD projects' target analyte list should be evaluated for poor performers.

Analytes that are in bold are also represented in Table 1 Standard Analytes.

.

Table 12

8260B CCV % Drift Criteria for Non-DOD Projects

Analyte	Cas No	CCV %D
1,1,1,2-Tetrachloroethane	630-20-6	30
1,1,1-Trichloroethane	71-55-6	30
1,1,2,2-Tetrachloroethane	79-34-5	30
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	50
1,1,2-Trichloroethane	79-00-5	30
1,1-Dichloroethane	75-34-3	30
1,1-Dichloroethene	75-35-4	20
1,1-Dichloropropene	563-58-6	30
1,2,3-Trichlorobenzene	87-61-6	40
1,2,3-Trichloropropane	96-18-4	30
1,2,4-Trichlorobenzene	120-82-1	40
1,2,4-Trimethylbenzene	95-63-6	30
1,2-Dibromo-3-Chloropropane	96-12-8	50
1,2-Dichlorobenzene	95-50-1	30
1,2-Dichloroethane	107-06-2	30
1,2-Dichloropropane	78-87-5	20
1,3,5-Trichlorobenzene	108-70-3	30
1,3,5-Trimethylbenzene	108-67-8	30
1,3-Dichlorobenzene	541-73-1	30
1,3-Dichloropropane	142-28-9	30
1,4-Dichlorobenzene	106-46-7	30
2,2-Dichloropropane	594-20-7	40
2-Butanone (MEK)	78-93-3	50
2-Chloroethyl vinyl ether	110-75-8	50
2-Chlorotoluene	95-49-8	30
2-Ethyl-1-Hexanol	104-76-7	50
2-Hexanone	591-78-6	50
2-Methyl-2-propanol	75-65-0	50
4-Chlorotoluene	106-43-4	30
4-Isopropyltoluene	99-87-6	30
4-Methyl-2-pentanone (MIBK)	108-10-1	50
Acetone	67-64-1	50
Acetonitrile	75-05-8	50
Acrolein	107-02-8	50
Acrylonitrile	107-13-1	50
Benzene	71-43-2	30
Bromobenzene	108-86-1	30
Bromoform	75-25-2	40
Bromomethane	74-83-9	50
Carbon disulfide	75-15-0	50
Carbon tetrachloride	56-23-5	30
Chlorobenzene	108-90-7	30
Chlorobromomethane	74-97-5	40
Chlorodibromomethane	124-48-1	40

Analyte	Cas No	CCV %D
Chloroethane	75-00-3	50
Chloroform	67-66-3	20
Chloromethane	74-87-3	50
cis-1,2-Dichloroethene	156-59-2	30
cis-1,3-Dichloropropene	10061-01-5	30
cis-1,4-Dichloro-2-butene	1476-11-5	50
Cyclohexane	110-82-7	30
Dibromomethane	74-95-3	30
Dichlorobromomethane	75-27-4	30
Dichlorodifluoromethane	75-71-8	50
Ethyl acetate	141-78-6	50
Ethyl ether	60-29-7	40
Ethylbenzene	100-41-4	20
Ethylene Dibromide	106-93-4	30
Hexachlorobutadiene	87-68-3	40
Hexachloroethane	67-72-1	40
Hexane	110-54-3	40
Iodomethane	74-88-4	50
Isobutyl alcohol	78-83-1	50
Isopropyl ether	108-20-3	30
Isopropylbenzene	98-82-8	30
Methacrylonitrile	126-98-7	50
Methyl acetate	79-20-9	50
Methyl tert-butyl ether	1634-04-4	30
Methylcyclohexane	108-87-2	30
Methylene Chloride	75-09-2	40
m-Xylene & p-Xylene	179601-23-1	30
Naphthalene	91-20-3	40
n-Butanol	71-36-3	50
n-Butylbenzene	104-51-8	30
N-Propylbenzene	103-65-1	30
o-Xylene	95-47-6	30
sec-Butylbenzene	135-98-8	30
Styrene	100-42-5	30
Tert-amyl methyl ether	994-05-8	40
Tert-butyl ethyl ether	637-92-3	30
tert-Butylbenzene	98-06-6	30
Tetrachloroethene	127-18-4	40
Tetrahydrofuran	109-99-9	50
Toluene	108-88-3	20
trans-1,2-Dichloroethene	156-60-5	30
trans-1,3-Dichloropropene	10061-02-6	30
trans-1,4-Dichloro-2-butene	110-57-6	50
Trichloroethene	79-01-6	30
Trichlorofluoromethane	75-69-4	50
Vinyl acetate	108-05-4	50
Vinyl chloride	75-01-4	20

Table 13

Summary of QC Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
BFB Tune	Prior to ICAL and at the beginning of each 12-hour period.	See Section 11.3	Retune instrument and verify. Rerun affected samples.
Minimum 5-point Initial Calibration, 6-point for quadratic curves (minimum 3-point Initial Calibration for Method 624)	Initial calibration prior to sample analysis	For Method 624: RSD for RFs: ≤ 35% for all analytes. For Method 8260B: 1. Average Response Factor for SPCCs: ≥ 0.30 for chlorobenzene, and 1,1,2,2-tetrachloroethane; ≥ 0.10 for chloromethane, bromoform, and 1,1-dichloroethane 2. RSD for RFs for CCCs: ≤ 30% For Method 8260C: 1. Average Response Factor for specified compounds: See table 14 2. RSD for all compounds: ≤ 20% **For DOD requirements above and one option below:** Option 1: RSD for each analyte ≤ 15% Option 2: Linear regression least squares regression r^2 ≥ 0.990 Option 3: Non linear least squares regression r^2 ≥ 0.990 and 6 points must be used.	Terminate analysis; correct the problem; recalibrate. Problem must be corrected. No samples may be run until ICAL has passed.
ICV or QCS	Following initial calibration.	Method control limits of all analytes under method 624	Terminate analysis; correct the problem; recalibrate.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		8260B: ±40% for non-DoD projects and ±55% for poor performers 8260C: ±30% for non-DoD projects and ±55% for poor performers **For DoD:** ±20% recovery and ±30% for poor performers with prior written approval	
Relative Retention Times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL. Laboratory may update RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (e.g. column clipping).
CCV	Daily before sample analysis and every 12 hours of analysis time.	8260B: 1. Avg RF for SPCCs: ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethene; ≥ 0.10 for chloromethane, bromoform, and 1,1-dichloroethane; 2. %D/Drift for CCCs ≤ 20%D. 3. %D/Drift for nonCCCs < limits in Table 12. 8260C: 1. Avg RF for specified compounds: See table 14 2. %D/Drift for 80% of compounds ≤ 20%D.	Correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all sample since the last successful CCV.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		For DoD: 1. Avg RF: see method 2. Opening CCV: %D/Drift for all target compounds and surrogates ≤ 20%D. 3. Closing CCV: %D/Drift for all target compounds and surrogates ≤ 50%D.	
Internal Standards (IS) verification	Every field sample, standard, and QC sample	Retention time ± 30 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard. **For DoD:** Retention time ± 10 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples while system was malfunctioning is mandatory.
Method Blank	One per batch of 20 field samples or fewer.	The result must be < RL or < 5% the amount measured in any sample or 1/10 the regulatory limit. **For DoD:** No analytes detected > ½ RL and > 10% the amount measured in any sample or 1/10 the regulatory limit. For common laboratory contaminants no analytes detected > RL.	Re-extract and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
LCS	One per batch of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported. QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	See Section 9.5 for additional requirements.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Surrogate	All field and QC samples.	Must be within laboratory control limits, unless it fails high and the sample is ND, or matrix interference is confirmed by a reanalysis or MS/MSD performed on the sample, or client specific requirements exist. **For DoD:** QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	See Section 9.4 for additional requirements.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported and must use LCS control limits. MD/MSD RPD ≤20%	See Section 9.5 for additional requirements.

Table 14

8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.060	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.020	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.020	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.060	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.060	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

b Data provided by EPA Region III laboratory.

Appendix A - Modifications for Method 624

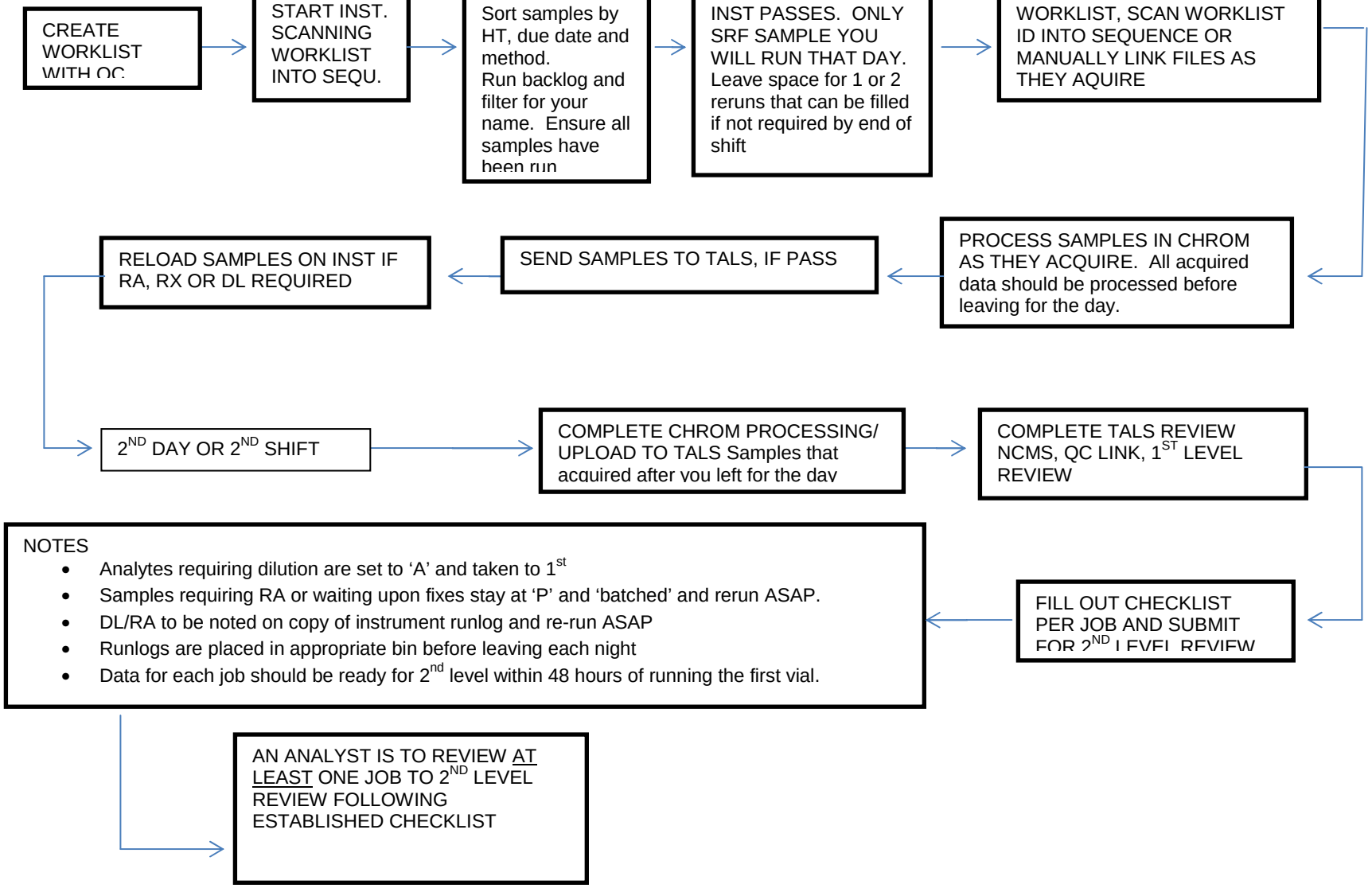
Requirements for EPA 624

1. Method 624 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
2. The tune period for this method is defined as 24 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 24 hour frequency.
3. The initial calibration curve for this method requires at least three points.
4. Sample concentrations are calculated using the average RRF from the initial calibration curve.
5. CCC evaluation is consistent with the published 624 CCC recovery criteria. Refer to method 624 for ranges.
6. Each target analyte is assigned to the closest eluting internal standard.
7. Initial demonstration of Proficiency
   - The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L.
8. Initial calibration curve requirements:
   - Target compounds must have RSD ≤ 35%.
   - If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.
9. Continuing calibration verification requirements:
   - The laboratory control standard is from a different source than the initial calibration standard. The daily CCAL concentration is 50 ug/L. The LCS concentration is 20 ug/L.
10. Matrix Spike and LCS Requirements
    - The matrix spike and LCS/LCSD are spiked at 20 µg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method.
11. Consistent with the other volatile methods, corrections for recovery are not allowed.
12. Qualitative Identification - The relative intensities of ions should agree to within ±20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.)
13. Method clarifications, modifications and additions
    - Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.
    - Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
    - The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including 1,4-dichlorobenzene-d4, which are not listed in Table 3 of the source method. Toluene-d8 is used as a surrogate compound, which is also not listed in the source method.
    - The lab is preparing internal standards at 10 ug/L and applying the same criteria designed for 30 ug/L in the Method. The lower the concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the

method. It could only be more challenging for the lab to meet the acceptance criteria at 10 ug/L; it provides a higher level of data quality.

- Method 624 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, we scan from 35 to 300 and include all ions needed for analysis.
- Method 624 describes dilutions “if response of any m/z” exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.13) is equivalent.
- Method 624 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta and para xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. Any development work to add compounds would take this into account.
- Method 624 has requirements for purge time and desorb conditions. Purge time for samples is 11 minutes ± 0.1 minutes at ambient temperature. After the 11-minute purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180℃ while b ackflushing the trap with an inert gas for four minutes.
- The purge gas flow rate (40 mL/min ± 5 mL/min) should be measured at the vent and recorded in the maintenance logbook.
- Method 624 requires the trap to be baked for 10 minutes prior to analysis.

Appendix B; PROCESS FLOW FOR SEATTLE VOA



Title: Determination of Volatile Organic Compounds by GC/MS Selected Ion Monitoring [Methods 8260B and 8260C]

Approvals

Signatures on File

Isaac Hooper Date
Volatiles Department Manager

Manjit Nijjar Date
Health & Safety Manager / Coordinator

Terri Torres Date
Quality Assurance Manager

Dennis Bean Date
Laboratory Director

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1.

1.2 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 2 µg/L for 8260B SIM and 8260C SIM waters, 5 to 70 µg/kg for soils. Reporting limits are listed in Table 1 and Table 2.

1.3 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer in selected ion mode, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol and an aliquot of the methanol is purged.

2.3 In the purge-and-trap process, an inert gas (generally Nitrogen) is bubbled through the solution at ambient temperature and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer in selected ion mode.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 Definitions

3.1 Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each 4-bromofluorobenzene (BFB) analysis will normally start a new batch. Batches for high-level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

The Quality Control batch must contain a matrix spike/spike duplicate pair (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. If there is insufficient sample to perform the MS/MSD, a duplicate LCS is used to establish batch precision when requested by the client. Refer to SOP TA-QA-0620 for further details of the batch definition.

3.2 Method Blank (MB) or Laboratory Reagent Blank (LRB)

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data. Sparged water or water that has been boiled then cooled to ambient temperature is used as the blank medium for water batches and muffled Ottawa Sand for soil batches. Prepared (muffled at 400C for at least 4 hours) batches of Ottawa sand are tracked using the reagent data base in the Laboratory Information Management System (known as TALS) and are at the time of the writing of this SOP named with the following convention: VoaSand_XXXXX.

3.3 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)

A blank matrix (reagent water or muffled Ottawa Sand) is spiked with the analytes of interest and is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.4 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) or Laboratory Fortified Sample Matrix (LFM)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6 Initial Calibration Verification (ICV) or Quality Control Sample (QCS)

The ICV is a second-source calibration verification standard. The QCS is reagent water or an environmental sample that is fortified with target analytes at known concentrations. This too is a second-source standard, i.e., different than the source of calibration standards.

3.7 Continuing Calibration Verification (CCV) or Laboratory Performance Check Solution (LPC)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, boiled and cooled to ambient or sparged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly Methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 The autosampler, purge and trap, gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 Cut resistant gloves or a protective cloth must be used when opening voa vials.

5.1.5 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.
- Gas Chromatographic Column used for 8260 SIM:

  : 60 m X 0.25mm ID DB-VRX with 1.4 µm film thickness.

Note: Other columns may be used. The serial number of the column used is documented in the instrument maintenance logbook.

- Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria.
- GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.
- Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.
- Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Mid-level waters, mid-level soils, and low level soils are purged directly from a VOA vial.
- Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax/Silica gel/Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.
- Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.
- Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.2 Computer hardware and software

- Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.
- LIMS system: TALS version 1.0 or higher.
- Data acquisition system: Agilent (Hewlett Packard) ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.
- Data processing: Chrom version 1.2 or higher. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.3 Supplies

- Microsyringes: 0.5 µL gas tight and larger, 0.006-inch ID needle
- Balance: Top-loading balance capable of weighing 0.1 g. The balance used for sample preparation is calibrated daily by a designated primary analyst (a back-up analyst is also assigned should the primary be unavailable). The analyst must perform this check according to SOP TA-QA-0014. It is also the responsibility of any analyst performing work on the balance to check the Balance logbook to determine if the daily calibration check has been completed, before beginning work.

- Scintillation Vials: 20 mL with screw caps.
- Volumetric flasks: 50 mL and 100 mL, class A with ground-glass or Teflon ® stoppers.
- Spatula: Stainless steel.
- Disposable pipettes: Pasteur.
- pH paper: Wide range (0-14) and narrow range (0-2.5).
- Helium: Ultra high purity, gr. 5, 99.999%.
- Nitrogen: Ultra high purity, from cylinders or gas generators, may be used as an alternative to helium for purge gas.

  Note: The use of Nitrogen as a purge gas is not allowed for analysis of VOA contaminants in drinking waters.
- Compressed air: Used for instrument pneumatics.

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Reagents

7.2.1 Methanol: Purge and Trap Grade, High Purity

7.2.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas for a minimum of 1 hour or boiling and cooling to ambient temperature prior to use. Other methods of preparing reagent water are acceptable.

7.3 Standards

7.3.1 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturer's expiration date.

7.3.1.1 Purchased standards are stored at the manufacturer's specifications (i.e. ambient, freezer, refrigerator). Standards prepared from these purchased standards are stored in the freezer.

7.3.2 Calibration Stock Standard Solutions: Components of stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at <-10°C. Each month a new standard is prepared. Note: Standards may be prepared on a more frequent basis based on analyst observed signs of degradation.

7.3.3 Calibration Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. Generally, an analysis of all individual compounds meeting the method criteria will suffice, however a continual failure of a compound may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem (two CCVs in a row fail), then a new initial calibration must be performed.

7.3.4 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

Likewise, medium level methanolic calibration standards are prepared in reagent water with a matrix matched methanol concentration of one fortieth P & T methanol using the secondary dilution standards and must be prepared daily.

7.3.5 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Table 4 for internal standard components.

7.3.6 Surrogate Standards: Refer to Table 5 for surrogate standard components and standard concentration.

7.3.7 Laboratory Control Sample Spiking Solutions: Refer to Table 6 for LCS components and standard concentration.

7.3.8 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 6.

7.3.9 Tuning Standard: A standard is made up that will deliver 5 ng of 4-Bromofluorobenzene on column upon injection.

7.4 As soon as standard preparations for the day are completed, the standards must be returned to the freezer.

7.5 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. The holding time for acid-preserved samples is 14 days from sample collection. For compliance with Method 8260B and 8260C, unpreserved samples must be tested within 7 days of collection.

8.2 Soils

8.2.1 Approved sampling containers for Method 5035 are EnCoreTM or TerraCoreTM and VOAs preserved with methanol.

8.2.2 Soil collected using the EnCoreTM or TerraCoreTM sampler must be preserved in the laboratory within 48 hours of sampling.

8.3 Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Three 40-mL VOA vials	40 mLs	HCl, pH < 2; Cool 0-6°C	14 Days	40 CFR Part 136.3 / 5030
Waters	Three 40-mL VOA vials	40 mLs	Cool 0-6℃	7 Days	40 CFR Part 136.3 / 5030
Soils	Three Encore Samplers	5 grams	Cool 0-6℃ or -10 to -20℃	48 Hrs for Preservation or 14 Days	5035A
Soils	40 ml VOA vial or 4oz septa top jar	10 grams or 25 grams	Methanol Cool 0-6℃	14 Days	5035A

[1] Inclusive of preparation and analysis.

8.4 Aqueous samples are stored in three 40 ml glass VOA vials with Teflon lined septa at 0-6oC, with minimal headspace. If a bubble is present and it is less than 6 mm in diameter, analysis may continue with the appropriate NCM added. If headspace exceeds this amount, then a non-conformance memo must be written and in some cases client approval requested to continue analysis.

8.5 Soil Sample Collection using Field Methanol Preservation

8.5.1 A pre-tared four ounce volatile soil jar with an accompanying VOA vial containing 25ml of a methanol/surrogate solution containing the surrogate TFT is sent out for each sample required when the sampling occurs in the state of Alaska. Otherwise a pre-tarred VOA vial containing 10 mls of methanol is sent out for each sample required. In addition the appropriate amount of trip blanks are also sent out. All bottles sent to the field are labeled with the tare weight and lot number of the methanol/surrogate solution or methanol.

8.5.2 For sampling in the state of Alaska, twenty-five gram field samples are collected by placing an appropriate amount of sample in the four ounce soil jar with septa lined lid and then adding one VOA vial containing the 25 mls of methanol/surrogate solution. Otherwise, ten gram field samples are collected by adding an appropriate amount of sample to a 40 mL VOA vial containing 10 mL of methanol.

8.6 Soil Sample Collection using EnCoreTM or TerraCoreTM samplers.

8.6.1 If the sample is collected a sample in an EnCoreTM sampler a minimum of one EnCores should be provided to the lab. If the samples are collected using the TerraCore sampler a minimum of one 40-ml VOA vial with 5 grams of soil. Samples must be received prior to 48 hours from sampling in order to be frozen or extracted in methanol. Following shipment back to the laboratory, the soil preserved in methanol.

8.7 A refrigerator or freezer blank is stored in each refrigerator or freezer with the samples. This is analyzed at minimum every 14 days, but may be analyzed more frequently as needed (see SOP TA-QA-0616). The refrigerator or freezer blank should be run immediately after the method blank.

8.8 Percent Moisture Correction for Soils

A percent moisture correction may be performed on soil samples to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect, as required by the client. Percent moisture must be determined if results will be reported as dry weight and percent moisture correction to be performed; refer to SOP TA-WC-0125 for determination of percent moisture. For all methanolic samples with a % moisture of greater than 10%, the following formula is used to determine the corrected final volume:

Corrected FV = ((g of sample * % Moisture/100) + mL of Methanol) * 40

(Also noted in section 12.9)

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in the TestAmerica Seattle QAM.

9.1.2 Specific QC requirements for Federal programs, e.g., USACE and Navy projects, are described in DoD QSM v5.1 or the latest promulgated version.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and may also come in the form of email or written notifications distributed at "project kick off" meetings.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument and in each analytical batch.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards and before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is muffled Ottawa sand. For high-level volatiles, the method blank consists of 10 mL of reagent grade methanol and ten grams of muffled Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: For DoD/LaMP projects the method blank must not contain any analyte of interest at or above one-half the reporting limit or above 1/10 the measured concentration of the analyte in the associated samples or 1/10 the regulatory limits, whichever is greater. For DoD projects, when written approval is received (method notes will contain “2CLC” or “Std Var App” to indicate approval has been received), the method blank must not contain any common laboratory contaminants above the reporting limit. Contamination up to the reporting limit is allowed for non DoD/LaMP projects or at or above 1/10 of the measured concentration of that analyte in the associated samples, whichever is higher.

The method blank must have acceptable surrogate recoveries.

Corrective Actions: For DoD projects, if the analyte is a common laboratory contaminant (i.e., acetone, 2-butanone, carbon disulfide and methylene chloride), and written approval has been received, the data may be reported with qualifiers if the concentration of the analyte is less than the reporting limit. For non-DoD if the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone, ethyl ether, Acetonitrile and hexane) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than the RL (less than one half the RL for DoD clients) in the samples associated with an unacceptable method blank, the data may be reported with qualifiers for non-DoD clients. For DoD clients the data may only be reported if written approval has been received.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all affected analytes in the associated samples are flagged as required by the project, and appropriate comments may be made in a narrative to provide further

documentation.

9.4 Surrogates

Every sample, blank (including instrument blanks), and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 5.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean or as defined by project or program requirements. Surrogate recovery limits are updated at a fixed frequency by QA and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

NOTE: For LaMP client samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the LaMP client representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

9.5 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard and the method blank, and normally before any samples. The LCS contains all of the analytes of interest (see Table 6) and must contain the same analytes as the matrix spike.

Acceptance Criteria:

The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data or based on project/program limits. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated at a set frequency by QA and are stored in the LIMS.

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAC. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

For non-DoD projects, if the LCS recovery is high and there are no detections in the associated samples for the affected analytes the data may be reported with qualifiers. For DoD projects the data may only be reported with qualifiers if approval has been received in writing (method notes will contain “3HR” or “Std Var App” to indicate approval has been received).

Note: For DOD projects, all exceedances of LCS Control Limits, are subject to corrective action. Therefore, all instances of LCS failures including the high bias but not detected in the associated samples scenario, must be investigated. For example and as noted above, the randomness of these failures can be evaluated or the spike solution can be re-verified. When the source of the problem is identified, corrective action is taken or variance from the QSM is requested.

Corrective Actions:

If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records (via NCMs and the case narrative) and in the final report. Examples of

acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 6. The selection of the spike solution is dependent on client program requirements. The matrix spike/duplicate must be analyzed at the same base dilution as the unspiked sample, even if the matrix spike compounds will be diluted out. Dilutions (beyond the base dilution if necessary) of MS/MSD analyses are not required unless there are specific client instructions to do so. If necessary, this requirement will be passed to the laboratory through the PM by means of the mechanisms described in section 9.1.3 of this SOP.

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated at a regular frequency by QA and are stored in the LIMS.

Corrective Actions: If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/matrix spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch, except in cases where a high bias is indicated and no target is detected above the reporting limit in any associated sample.

If an MS/MSD is not possible due to limited sample, then a LCS duplicate (LCSD) should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 Internal Standards

Internal standards are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard recoveries are monitored to verify that instrument performance is acceptable. Criteria for calibration standards are delineated in Section 10. If the internal standard data indicate instrument failure, the samples may require reanalysis. If not, the impact on sample data is evaluated and the data is flagged appropriately.

Acceptance Criteria: Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in internal standard response is a matrix effect confined to an individual

sample, reanalysis is not necessary. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.8 If batch QC samples or trip blanks are re-analyzed to confirm a recovery or result, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (i.e. no internal standard added, bent autosampler needle, etc).

9.9 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

10.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.0 Calibration

11.1 Summary

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of a separate calibration is required for low level water.

11.2 Recommended Instrument Conditions

11.2.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 seconds/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute (± 5 mL/min)
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

11.2.2 Gas Chromatograph Temperature Program

The temperature programs vary with the column type and instrumentation used. The GC run program on each instrument should be optimized so that each peak is broad enough to accommodate at least 5 scans across the peak (not counting the scans at the baseline start and end of the peak). The actual individual method parameters used are stored in each individual instrument methods folder on the network and can be referenced there.

11.2.3 Mass Spectrometer/ Scan Parameters

The mass spectrometer is operated in selective ion monitoring mode, with the following ions monitored in each window. The start times for each window are representative, and may vary slightly with chromatographic maintenance.

Scan Window	Masses Monitored (z)	Start time (min)	Scan Rate (Scans/Sec)
1	39,53,54,62,64,94,96	2.00	1.57
2	45,46,59,61,65,96,98	5.80	1.41
3	62,64,65,67,83,85,98,111,113	8.00	1.23
4	47,60,77,78,81,83,85,95,96,132,146,174	9.10	0.92
5	39,43,49,58,61,75,79,81,85,97,98,100, 107,109,110,127,129,131,164,166	10.20	0.55
6	82,117,131,133,171,173,175	12.25	1.41
7	50,61,75,83,85,95,96,110,131, 174,175,176,177	13.26	0.85
8	75,111,115,146,148,150,152	14.40	1.41
9	102,118,127,128,190,223,225,227	16.90	1.25

11.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed below and in Table 7 for a maximum of a 5 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB. It is critical to accurately estimate the number of samples that can be analyzed within the 12-hour window. When a tune isn't analyzed every 12-hours (i.e., samples are analyzed outside of the 12-hour window), the event must be documented in a non-conformance memo and corrective must be taken. Whenever feasible, samples that were analyzed outside of the 12-hour window will be re-analyzed within a new 12-hour window. When reanalysis is not feasible, results for the affected samples can only be reported if it's technically justified (e.g., subsequent tune passes), the data has been qualified, and it's been authorized and accepted by the client. The BFB may be taken from a specified full-scan BFB Tune injection or from the CCVIS from a SIM injection. In the case of a calibration sequence, a specified BFB Tune must be injected prior to the injection of the first calibration standard. If an acceptable tune is not achieved, the autosampler prepares another tune standard by adding BFB to either the CCVIS or an instrument blank. The autotune process is repeated once. If the subsequent tune attempt fails, one or more of the corrective actions suggested in the TestAmerica, Inc. corporate tune policy, CA-Q-QM-002 are attempted.

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	50 to 120 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

11.4 Initial Calibration

11.4.1 A series of five or more initial calibration standards is prepared and analyzed for the target compounds. Nominal calibration levels are 0.005, 0.01, 0.05, 0.1, 0.5, 1, 5, 10, and 25 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Table 3 shows the calibration levels for each analysis. The purge volume is 5 mL. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements. Calibration levels may also vary based on analyst discretion in so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the lowest point on the curve is at or below the current TestAmerica Seattle reporting limit.

11.4.2 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

11.4.3 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

11.4.4 Rejection of Calibration Points

Calibration levels below the reporting limit may be removed provided that the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves), and the lowest standard is at or below the TestAmerica Seattle reporting limit.

High point calibration levels may also be removed so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the midpoint of the curve (the ICIS) is not the highest point of the calibration range.

Generally, it is NOT acceptable to remove mid-points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the maintenance logbook. Then the calibration standards must be reanalyzed. If, however, there is documented evidence of a problem with a calibration point (e.g. misinjection, poorly sealed vial, etc…) then one point might be rejected, but it is recommended to re-calibrate.

Refer to Corporate SOP GA-Q-S-005, Calibration Curves, for further details.

11.4.5 Internal Standards

Internal standard calibration is used. The internal standards are listed in Table 4. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Corporate SOP CA-Q-S-005, for calculation of response factor and other related algorithms.

11.4.6 The most common target analytes are checked for a minimum response factor for each calibration level. See Table 10 for the compound list and minimum relative response factor criteria. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. For any analyte non-detect associated with a calibration that fails the minimum response factor criteria there must be a demonstration of adequate sensitivity at the quantitation limit. This is achieved by the successful analysis of a CCVL (CCV at the reporting limit) in the same analytical batch. The criterion for the CCVL is detection only but the standard qualitative identification criteria in the method must be met.

11.4.7 If all of the %RSD values in the calibration are ≤ 15% for 8260B, then all analytes may use average response factor for calibration. If all of the %RSD values in the calibration are ≤ 20% for 8260C, then all analytes may use average response factor for calibration. For analytes that fail the RSD criteria, use linear or quadratic curve.

11.4.8 If the software in use is capable of routinely reporting curve coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD ≤ 15% (8260B or DoD) or ≤ 20% (8260C) for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve, then the appropriate curve should be used for quantitation. The correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.9 If the software in use is capable of routinely reporting curve coefficients for data, then calibration on a curve must be used for all analytes with %RSD > 15% (8260B) or %RSD > 20% (8260C). The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.10 For 8260C no more than 10% of compounds can fail the 20% RSD/0.990 correlation coefficient requirement. Any individual analyte result that fails the 20%RSD/0.990 correlation coefficient requirement must be flagged or narrated as an estimated concentration.

11.4.11 For any analyte non-detect associated with a calibration that fails the 20%RSD/0.990 correlation criteria there must be a demonstration of adequate sensitivity at the quantitation limit. This is achieved by the successful analysis of a CCVL in the same analytical batch. The criterion for the CCVL is detection only but the standard qualitative identification criteria in the method must be met.

11.4.12 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

11.4.13 Initial Calibration Verification (ICV)

Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. The ICV is generally run at 1 ug/L. As the ICV concentration must not be equal to or greater than the highest calibration level, other ICV levels than those

previously listed may be used or multiple levels of ICV may be needed to validate all compounds in the initial calibration curve.

For DoD and LaMP projects each target compound in the ICV must be <20% drift when compared to the initial calibration. For non-DoD and non-LaMP projects, all compounds must be <30% drift when compared to the initial calibration, except for poorly performing compounds listed in Table 8, which must be <45% drift.

For DoD projects analyses may continue if the drift is <30% for identified poor performing analytes with written approval from the client. (Method comment 4PP). See table 8 for the list of indentified poor performers.

For non-DoD and LaMP projects analyses may continue for those analytes that fail the criteria with approval from the client and an understanding that these results would be considered estimates and could be used for screening purposes.

Corrective Action: If the % drift falls outside acceptance criteria, assess the system for possible problems (standard degradation, etc.), re-prepare the verification standard and re-analyze. If the second ICV also fails, corrective action is required (e.g. system maintenance, re-preparing intermediate standards, etc.) and the calibration must be re-prepared and re-analyzed. An acceptable ICV must be achieved before sample analysis. No samples may be run until calibration has been verified. Analytes which do not meet the ICV % drift criteria will be removed from the calibration in the Chrom chromatography software for the method in which the analyte did not meet the method criteria to prevent the reporting of analytes using a deficient calibration curve.

11.4.14 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 11.5.

11.5 Continuing Calibration

11.5.1 The initial calibration must be verified every twelve hours. For DoD projects the calibration must also be verified with a closing continuing calibration standard (CCVC) at the end of each analytical sequence.

11.5.2 Continuing calibration begins with analysis of BFB as described in Section 11.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 5 calibration standard is generally used as the CCV and CCVC.

11.5.3 For non-DoD projects the RF data from the standards are compared with the initial multi-point calibration to determine the percent drift of the target compounds. For non-DoD and LaMP projects the % drift limits of target compounds should be <20% before analysis proceeds, except for poorly performing compounds listed in Table 8, which must be <45% drift.

11.5.3.1 Non target compounds that exceed the specified limits for Drift or Difference should be flagged.

11.5.4 For DoD and LaMP projects, the percent difference or drift (%D) of target and surrogate compounds must be within ± 20% for the opening CCV.

11.5.5 For DoD projects the percent difference or drift (%D) of target and surrogate compounds must be within ± 50% for the closing CCV (CCVC).

11.5.6 For non-DoD projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds (0.5 min) when compared to the most recent multi-point calibration. For DoD projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 10 seconds when compared to the most recent multi-point calibration. The 30 second criteria may only be used for DoD projects if prior written approval is received (method notes will contain “8ISRT” or “Std Var App” to indicate approval has been received). The internal standard

areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent multi-point calibration.

11.5.7 If target and/or surrogate compounds do not meet the criteria in Sections 11.5.3 through 11.5.8, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration. For non-DoD and LaMP, if two CCVs in a row fail, a new initial calibration must be performed. For DoD two additional consecutive CCVs must be run immediately. If both pass, samples may be reported without reanalysis. If either fails corrective action and re-calibration is required.

Corrective Actions:

For non-DoD projects if the CCV recoveries of the target or surrogates compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias. For DoD projects, if the CCV recoveries for target compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias only if prior approval has been received in writing (method notes will contain “3HR” or “Std Var App” to indicate approval has been received). For all other cases, results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

11.5.8 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

11.6 Sample Preparation

Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014

11.6.1 Sample extraction using in house extraction.

11.6.1.1 When extracting the sample, extrude (for 5 g EnCore™) or weigh (for 10 g in house soil extraction) the sample into a tared 20 mL scintillation vial. EnCore samples should be extruded into methanol upon receipt. Record scintillation vial lot number, the sample weight, the methanol lot number, the preparation date and the analyst who prepared it in the 5035 preparation batch in TALS. Sample weights are calculated in the laboratory by adding the tare weight of the scintillation vial to the “Tare Weight” column and then entering the weight of the scintillation vial plus sample into the “Vial & Sample” column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples were preserved in MeOH at another TALS lab the calculated initial sample weight can be manually entered into the “Initial Amount” column of the preparation batch sheet for the corresponding sample container ID.

11.6.1.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are also extracted. To prepare the method blank (MB); add 10-mL of methanol to 10 g Ottawa sand. To prepare the blank spike (LCS)/blank spike duplicate (LCSD), add 8 uL of the 8260 Working Solution and 10 mL of methanol to 10 g muffled Ottawa sand. (NOTE: The same metal spatulas to weigh soil samples must be used for measuring out the Ottawa sand).

11.6.1.3 If sufficient sample is available, one matrix spike (MS)/matrix spike duplicate (MSD) pair is extracted per extraction batch of up to 20 samples. If the sample set being extracted consists of products, waste oils, or other sample matrixes which based on analyst experience and discretion would not yield acceptable spike results due to matrix effects, a matrix duplicate (MD) and/or MD/MS may be prepared in lieu of and MS/MSD. To prepare the matrix spike (MS)/matrix spike duplicate (MSD), add 8 uL of the 8260 Working Solution and 10 mL of methanol to 10 g pre-weighed soil samples.

11.6.1.4 Add 5 mL (for EnCore™ or TerraCore™) or 10 mL (in house soil extraction) of methanol to all vials immediately after recording sample weight.

11.6.1.5 Vortex the samples for the extraction batch for approximately 10 to 30 seconds to break up any large clumps in the extraction vials. This is especially important for extruded samples as they may be compacted in the EnCore™ or TerraCore™ sampling device and come out as a pellet. If after 30 seconds a pellet still remains, vortex for an additional 30 seconds. If pellet still remains, further vortex mixing is not recommended, proceed to next step. It should be noted that the MB and LCS must be vortex mixed the same amount of time as the longest associated sample.

11.6.1.6 After all samples have been vortex mixed, place all samples for the extraction batch into shaker box and set timer for five minutes. It is recommended that the caps of all vials are checked and tightened before placing in shaker box to prevent leaking. If samples are present which still contain pelletized sample after vortex mixing in step 11.6.1.5, set the timer for 10 to 15 minutes. It is not recommended to shake samples for more than 15 minutes. If a sample still contains pelletized sample after shaking for 15 minutes, vortex the sample for an additional 15 seconds and shake entire batch for additional 5 minutes. Any pelletized sample remaining after second shaking is noted in and NCM in the extraction batch.

11.6.1.7 After samples are shaken, place samples four at a time into centrifuge with inserts for scintillation vials. Spin samples for a sufficient time to create a transparent but not necessarily uncolored layer of methanol extract above the extracted material. The time will vary depending on the nature and particle size of the extracted material. Three to five minutes at 50% speed is usually sufficient. Again it should be noted that the MB and LCS must be centrifuged at the same rate and for the same time as the longest centrifuged associated sample.

11.6.1.8 Solid extracts are stored in the scintillation vial used for extraction and are stored at 0-6°C. The extracts are removed from cold storage and are allowed to return to ambient temperature prior to analysis.

11.6.2 Sample extraction for field preserved.

11.6.2.1 Each containers tare weight is recorded in the TALS preparation batch. Most containers will contain a bar code with the tare weight information that can be scanned for automatic entry into the tare weight entry field in the preparation batch. Sample weights are calculated in the laboratory by adding the received weight of the sample jar to the “Vial & Sample” column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples received are in 4 oz jars with 25 mls of methanol (AK samples) the calculated initial weight of the sample must be adjusted to correct for

the weight of the methanol which is not included in the container tare weight. TALS will perform this calculation, however the analyst must enter a "1" into the "Tare Incl MeOH" column of the preparation batch sheet for the corresponding sample container ID.

11.6.2.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are prepared by the laboratory prior to sample analysis. A MB and an LCS sample consists of 10 g of muffled Ottowa Sand added to a scintillation vial followed by 10-mL of methanol.

11.6.2.3 Add the correct amount of matrix spiking solutions to all LCS samples. An aliquot of 8 uL of the 8260 Working Solution is added to 10 mL extracts. The addition of the spike solutions introduces a slight error, which can be neglected in the calculations.

11.6.2.4 The MB, LCS and any received samples which appear to contain "clumps" of sample which could be broken up with agitation are vortex mixed for up to 1 minute. If no samples require agitation, only the MB and LCS are vortex mixed for approximately 15 seconds to ensure mixing of the sand and added solutions.

11.7 Sample Analysis

11.8 Preliminary Evaluation

11.8.1 Sample screening

Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories. Refer to section 11.15 for Dilutions.

11.9 Sample Analysis Procedure

11.9.1 All analysis conditions for samples must be the same as for the continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.9.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD (if sufficient sample volume allows), an LCS, and a method blank.

11.9.2.1 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.9.2.2 It is not necessary to reanalyze batch QC (except for the method blank) with reanalysis of samples. However, any re-runs must be as part of a valid batch.

11.10 Water Samples

11.10.1 All samples and standard solutions must be at ambient temperature before analysis.

11.10.2 For standard level analysis 5 ml are sub sampled by the autosampler.

11.10.3 For standard level analysis internal standards and surrogates are added by the autosampler. Refer to Tables 4 and 5.

11.10.4 MS/MSD samples are prepared by injecting the 8260 Working Solution through the VOA vial septa using a bevel tipped syringe or directly into the MS or MSD VOA vial.

11.10.5 Purge the sample at ambient temperature with a trap temperature of 25OC.

11.10.6 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.10.7 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 1 minute or 2 minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260OC, and bake temperature is 220 to 270OC.

11.10.8 Immediately after analysis or immediately after opening the sealed VOA vial and obtaining the necessary aliquots for dilutions, the analyst must check the pH of aqueous samples with narrow range pH paper to ensure the pH is < 2. Record the pH of aqueous samples and the lot number of the pH paper used on the analytical batch sheets along with any dilution factors used. In those cases where the pH is > 2, initiate a non-conformance report and qualify the data, noting if the sample(s) was analyzed outside of the shortened seven-day hold time or zero-day hold time for aromatics.

11.11 Methanol Extract samples

11.11.1 Fill a VOA vial with reagent water, and remove 900 uL of water using a volumetric pipette.

11.11.2 Add 1075 uL of methanolic extract to the vial and immediately cap the VOA vial invert the vial to ensure that no air bubble larger than 4 mm is present. If there is an air bubble and it is greater than 4 mm, re-prepare sample.

11.11.3 The final volume of reagent water and methanolic extract used is entered into Chrome which then is uploaded into the analytical batch in TALS (43 mls).

11.11.4 As with water samples, internal standards and surrogates are added by the autosampler. Refer to Tables 4 and 5. TFT may be also be added to the field methanol (Alaska samples), thus the additional amount of TFT should be taken into account in the prep batch for AK samples.

11.11.5 Load the sample in the autosampler and proceed to analyze.

11.11.6 MS/MSD samples for in house extracts are prepared at time of extraction and are prepared for analysis as above. For field preserved samples, an in house post spike of the prepared sample is necessary, and is prepared by injecting the 8260 Working Solution through the VOA vial septa using a bevel tipped syringe.

11.11.7 Dilutions of methanolic extracts are made by adding proportional amounts of methanol extract to a new VOA vial. Samples of greater than 5x do not require the removal of water from the VOA vial prior to addition of methanolic extract.

11.11.8 Purge the sample at ambient temperature with a trap temperature of 25OC.

11.11.9 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.11.10 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 0-1 minute, bake time is 6 to 9 minutes, desorb temperature is 195 to 260OC, and bake temperature is 220 to 270OC.

11.12 Initial Review and Corrective Actions

11.12.1 Retention Times

For 8260B if the retention time for any internal standard in the continuing calibration changes by more than 30 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

For DoD projects of 8260C if the retention time for any internal standard in the continuing calibration changes by more than 10 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.12.2 Internal Standard Response

If the internal standard response in the daily continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

The internal standard response in a sample is compared to the internal standard response in the daily continuing calibration. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary, though may be required to prove matrix effect. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected. If the ISTD response falls below 50% and the sample has no target analyte detections above the RL (1/2 the RL for DoD projects), the data may be qualified and reported. This should be done in consultation with the PM and client, an NCM must be written.

11.12.3 Surrogate Standard Recoveries

The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect (this may be demonstrated by a MS/MSD analysis as well). Reanalysis at a dilution should be considered if appropriate. If a diluted analysis is necessary and surrogate recoveries are in control or less affected, this is sufficient to demonstrate matrix interference.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

11.13 Dilutions

Dilutions for waters are made directly into a 40mL VOA vial and should be prepared just prior to the GC/MS analysis of the sample. For example, a 10X dilution is made by filling a VOA vial to slightly overfull and then removing 4.3 mL of reagent water from the vial. 4.3 mL of sample is then transferred to the vial to bring it back to full and it is immediately capped for analysis. In the worklist thjs is recorded at a 10.0 dilutiuon,.

Soil dilutions are prepared by adding a smaller aliquot than the standard 1.075mL or by serial dilution for aliquots smaller than 20uL or viscous samples. In the prep batch this is recorded as the amount added to 43 mL. The worklist is left as a 1.0 dilution.

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample/extract is prepared and analyzed. A dilution should be prepared to ensure that the

majority of compounds being diluted for fall in the middle to upper part of the calibration curve (i.e. from 40-90% of their respective calibration range). All reported dilutions must be within the calibration range of the respective analytes and should be compared to other dilutions to ensure that the diluted data "makes sense" as a check for possible dilution errors. If this cannot be accomplished with a single dilution, multiple sample dilutions are necessary. Dilution levels should be considered carefully and it is recommended that a "complicated" sample (one which has more than three compounds which require dilution) is discussed with another analyst or area supervisor as necessary in order to minimize the number of dilutions required. Samples may be screened to determine the appropriate dilution for the initial run or historical site data may be used to determine initial dilutions.

11.13.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment and reasonable client requirements and requests.

11.13.2 Reporting Dilutions

The most concentrated dilution will be reported as the base dilution. Other dilution levels will report only the required diluted compounds and all other compounds and surrogates in the dilution will be set to acceptable in the LIMS system and not reported. Other reporting techniques may be required by specific project requirements or client request and will be transferred to the laboratory by the PM using the mechanisms previously discussed. See Section 9.1.3.

11.13.3 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP TA-WC-0125 for determination of percent moisture.

11.14 Instrument Maintenance

11.14.1 Agilent 5973 Inert, 5973 Network, 5975, 5975B, and 6890N

11.14.1.1 All circuit boards and peripheral attachments are dusted and vacuumed of debris and all plumbing and electrical connections inspected and adjusted; any replacement of worn parts is also done at this time.

11.14.1.2 The injection port is cleaned of debris by removing the injector and column nut and forcing clean methanol through the top of the injector into a waste container at the bottom. The inlet liner and gold seal are replaced as needed (e.g. visibly dirty or discolored, active site issues, etc.) The ion source is disassembled, cleaned, and reassembled with new filaments and insulators, if needed.

11.14.1.3 If low level soils cause recoveries for the internal standard to report low or purge flow to decrease in any consequent CCV's, the pencil filters or injector needle could be clogged. Methanol rinses of these parts may solve the issue, or replacing the part.

11.14.2 Column installation is performed when the following conditions are encountered;

- Heavy column bleed that cannot be eliminated by thermal conditioning.
- Loss of early eluting peaks due to column cutting.
- Inability to chromatographically resolve method performance compound peaks.
- Distortion of peak shapes i.e.; broadening, ghost peaks, split peaks that can't be resolved by injection port maintenance or flow control.

11.14.2.1 Turn the GC oven off and let the system cool to room temperature. Remove the column nut, liner, septum, and press tight inlet connector. Dispose of old column appropriately.

11.14.2.2 Cut approximately six inches off of the end of new columns. Install new column using appropriate sized ferrules and nuts.

11.14.2.3 Turn the GC on and set the injector temperature to 230 ℃, oven to the manufacturers recommended isotherm temperature or 10℃ below the manufacturers max temperature if an isotherm is not provided and condition for five minutes.

11.14.2.4 Perform an air water check on the system. When the air water spectrum shows acceptable levels, proceed with the mass calibration procedure. For additional information of column replacement see the manufacturer's operator's manual.

11.14.3 OI Analytical 4560 and 4660 Thermal Purge and Trap Concentrator Units

11.14.3.1 A new Supelco K-trap (Vocarb 3000) is installed and conditioned by stepping the unit through to the bake setting. A new trap should be conditioned for a minimum of one hour prior to use, but an initial conditioning of overnight is recommended as time allows.

11.14.3.2 Sample lines, internal valves, sparge cells, and sparge cell mounts and fittings are rinsed with purge and trap grade methanol or replaced as necessary.

11.14.3.3 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.14.3.4 The purge gas flow rate (40 mL/min ± 5 mL/min) should be measured at the vent and recorded in the maintenance logbook.

11.14.4 Archon 2000 or equivalent type auto sampler.

11.14.4.1 Remove debris and perform a calibration per manufacturer's instructions.

11.14.4.2 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.14.4.3 The guide rails are wiped down with a Kim wipe and n-Propanol to remove grease buildup and debris.

11.14.4.4 The syringe is cleaned per manufacturer's instructions to remove debris.

11.14.4.5 The rinse reservoir is refilled purge water prior to analysis.

11.14.4.6 The Internal Standard/Surrogate Standard vial is refilled as needed and primed after filling to ensure the instrument is drawing volume.

11.14.5 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the source and replacing the multiplier. In addition, a new initial calibration may be necessary if a large amount of routine maintenance occurs at once. This will be determined by evaluating the system using a CCV.

11.14.6 Maintenance Logbook

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service".

Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, tune, etc as may be the case).

If columns were replaced during maintenance procedures, the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook.

11.15 Troubleshooting

11.15.1 QUESTIONS TO ASK YOURSELF

11.15.1.1 MECHANICAL ISSUE?

- Has the problem just started, or has it been getting worse with time?
- Has anything recently been done to the instrument that may have caused the problem to start?
- Have there been any nasty samples run on the instrument recently?

11.15.1.2 ANALYTE ISSUE?

- Are there any other compounds out or trending out?
- Did the CCV and/or LCS show similar trends?
- Is this a one time event? Or did you see similar signs yesterday?
- Is it failing on other mass specs?
- Did anything change from the last time it passed?
- Have there been any nasty samples run on the instrument recently?

11.15.1.3 INTERNAL/SURROGATE ISSUE?

- Have they been stable up until now?
- Is the gas on and are the standards fresh?
- Are the glass vials correctly positioned and tight?
- Do they look like they are trending in any direction or just fluctuating?
- Is the tune stable? Are the ratios close to the ICAL tune?

11.15.2 BEST PRACTICE

11.15.2.1 If you can – replace the most likely piece in the system, label the old one with which instrument it's from, the date out and possibly the problem you are having e.g. R1, 071010 Bromoform. Test if the problem is fixed. If not, move onto the next thing and replace until the problem is fixed. Then work backward and reinstall the old pieces which are still OK. If you no longer need a part that is still good, label it as such. If a piece is bad and not even good for testing with throw it out and tell your department manager what piece and part number.

11.15.3 TYPICAL FIRST THINGS TO LOOK AT AND TRY

11.15.3.1 CHECK YOUR FLOWS (For soil mode, purge on archon)

- Set the Archon to purge for 111mins instead of 11mins then set a vial to purge and "pause" the concentrator. This will give ample time to troubleshoot.
- Use the flow meter to check the flow out the purge vent using a small clear tube as an adapter – you should have about 40mls.
- Take the transfer line off at the entrance to the concentrator and measure the flow – you should also have about 40mls.

- Generally this would suggest you don't have a blockage or a leak, but check the following first
- Find a clear tube that has been blocked at one end, place this on the purge vent while the vial is purging and establish the following:
- Did the bubbles stop straight away <2mins?
- Yes – the leak is in the Archon
- Have the bubbles stopped over a 5 – 7 min period?
- Yes – Most probably not a leak but could be a blockage (which includes the tube you put on the vent)
- No – There is a leak somewhere, most likely the concentrator.
- If there is no soil valve attached then remove the transfer line inlet (to concentrator) fitting and replace with a capped fitting, Run the concentrator full leak check.
- If all of the above passes then you do not have a problem with the flows/blockage/leak.
- Remember to set Archon back to 11mins purge and bake off trap before running again.

11.15.3.2 IF YOU HAVE A LEAK

- In the Archon – purge a vial and leak check around the top of the needle, the transfer line and the pencil filter in the back of the Archon.
- In the concentrator – with a vial purging leak check around all the fittings starting with the entry from the transfer line, the 4-port valve, the 6-port valve ad around the water management and trap.
- You can also check the flows coming out of the 4-port, leaving the 6-port, there is an adapter which can be added instead of the trap to measure through the water management, from the trap. If all of these measures around 40mls and still you have less than 37 coming out the purge vent it could be leaking across the valves in the side of the concentrator.
- REMEMBER – any Archon that has been changed to Nitrogen will not be able to be checked with the leak detector.

11.15.3.3 COMMON PLACE TO LOOK IF IT'S A ANALYTE PROBLEM

- **1,1,2,2-Trichloroethane RRF** – Could be any of the lines as often an active site. Start with transfer line, lines in concentrator, water management and trap, soil valve if installed.
- **Bromoform** – Flow is not 40mls, water management, transfer line, small lines in concentrator. This can sometime be a tune issue as well if it is somewhat different from normal.
- **Napthalene** carryover/low response – Often water management, change this out and condition with 5 or 6 varying concentrations of an old main or 524 std. e.g. 20ppb, 80ppb, 10ppb, 50ppb etc. Could also be transfer line.
- **Vinyl Chloride** – Usually the first compound to fail low in the standard mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **1,1-Dichloroethene** – Usually the first compound to fail low in the Main mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **Chloroform** – usually your tune is differing from the ICAL
- Contamination – look back at previous runs, if you have high hits for this compound then run 20 – 30 blanks to clean it up. If you still have high concentrations then the sample possibly blew back into the instrument so could be purge valves or the pencil filter in the back of the Archon.

11.15.3.4 PEAK SHAPE DIFFERENT

- If you have peak tailing on the front end compounds such as Vinyl Chloride, Chloromethane etc then it is often a result of a worn trap.

11.15.3.5 SURROGATES FAILING

- If you have 1,2-DCA failing and all the other surrogates are OK, then the likely cause is the tune. Often the ratios will have moved. Follow the surrogates, if low, then drop ratios etc
- If you have all the surrogates out and it seems to be consistently that way, it could be the following,
  - Is the gas turned on?
  - Is the surrogate vial positioned properly on the archon
  - Your concentration levels in the ICAL maybe incorrect if you have just ICALed.
  - The valve has perhaps worn a little and the nominal value in target needs to be updated, but this can only happen after an ICAL.
  - If the surrogates need to be weighted linear or linear in an ICAL and you see them trending this way for a while its possible the column has gone bad or you may have a standard valve leak.
  - You can check to see if it's the archon causing the problem by manually injecting IS/SS and turn off the IS and SS on the archon
- If two surrogates are high and two are low, sometimes this is because your Archon surrogate is not matching with the surrogate you ICAL'd with. Try swapping out the Archon surrogate and if this does not fix it, re-ICAL the Supp with the new surrogate.

11.15.3.6 INTERNALS FAILING, TRENDING OR BOUNCING

- How fresh is your internals? If they are more than say a month old, they may have gone bad. Is the vial on properly and done up tight (not too tight).
- Is the gas turned on?
- How long has the instrument been using the current filament? More than 4weeks then you may want to change to the other or change the filaments if they are both spent.
- If the internals bouncing seems to correlate with the tune then most likely you want to change filament or clean the source.
- If the internals fall to a value and sit there or raise to a value and sit there you may want to consider ICAL it at that tune setting, instruments have a kind of sweet spot.
- Internals falling off maybe a water buildup and the parameters need looking at on the concentrator. Have a look at other instruments to see how they are set up and try one or two different settings to establish if this helps. Always keep a note of where they were at and what you have changed them too. Best place to record this is on your sequence and in the white folder under daily changes.
- Split ratios can also effect internals, try increasing the ratio to more like 45:1 if not already at this level.

11.15.3.7 CONCENTRATOR STOPS

- Turn it off and on again, it is common for the concentrator to lose the plot every so often.
- Shows a temperature error – check to make sure the trap is covered and the main cover is down properly, any heat escape will cause an error.

11.15.3.8 SEQUENCE STOPS

- You have started on a file that already exists and you have not checked the overwrite file box.
- Communication problem with the GC and MS. Turn off the MS and GC and restart the computer. Restart the MS and GC.

11.15.3.9 ARCHON STOPS

- Standard home errors – syringe already home – use the maintenance menu on the archon to step you through cleaning the syringe. Before putting back together smear the inside of the syringe with 'Nose grease'.
- Standard home errors -

12.0 Calculations / Data Reduction

12.1 Qualitative Identification for SIM Analysis

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1.1 The sample component retention time must compare to within ± 0.06 RRT units of the retention time of the standard component. For reference, the standard must be run within the same twelve hour tune as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.1.3 All data are subject to two levels of technical review, as described in SOP TA-QA-0635.

12.2 Accuracy

ICV / CCV, LCS % Recovery = observed concentration / known concentration x 100

MS % Recovery = (spiked sample) - (unspiked sample) / spiked concentration x 100

12.3 Precision (RPD)

Matrix Duplicate (MD) = |orig. sample value - dup. sample value| / [(orig. sample value + dup. sample value)/2] x 100

12.4 Response Factor (RF)

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng.
C_x = Concentration of the compound being measured, ng.

12.5 Standard deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of Xi.

12.6 Percent relative standard deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

12.7 Percent drift between the initial calibration and the continuing calibration:

$$\%Drift = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100\%$$

Where:

$C_{expected}$ = Known concentration in standard.
C_{found} = Measured concentration using selected quantitation method.

12.8 Calculation of Results for Waters

Sample Conc (ug/L) = C x D

Where:
C = On Column concentration (ppb)
D = Dilution Factor

12.9 Calculation of Results for Methanol Extracts

Sample Conc (ug/Kg) = [On Column (ug/L)] x Extraction Final Volume (mL) x VOA Vial Volume (mL) x 1L (CF) x 1000g (CF)
Amount of Soil Sample (g) Amt of MeOH Extract (mL) 1000mL 1Kg

VOA vial volume is 43 ml and when the extract doesn't require a dilution, 1.075 mL of the methanol extract is used. So, the equation becomes:

Sample Conc (ug/Kg) = [On Column (ug/L)] x Extraction Final Volume (mL) [1] x 43 (mL) x 1L (CF) x 1000g (CF)
Amount of Soil Sample (g) [2] 1.075 (mL) 1000mL 1Kg

[1]Extract Final Volume, miscible solvent corrected (mL) = ((g of samples * % moisture/100) + ml of MeOH) * 40
(used when % Moisture of the soil sample is greater than 10%).
[2]Amount of Soil, dry-weight corrected (g) = sample mass (g) * (100 - % moisture/100)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

Note: For all methanolic samples with a % Moisture of greater than 10%, it is necessary to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect. This is done by the following equation:

Corrected FV = ((g of samples * % moisture/100) + ml of MeOH) * 40

In these situations, an "other observation" NCM must be generated and the correction and above formula must be noted in the case narrative.

12.10 Upon completion of the analytical sequence:

12.10.1 Review chromatograms online and determine whether manual data manipulations are necessary.

12.10.2 Manual Integrations

12.10.3 All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

12.10.4 Manual integrations may be processed using Chrom, which stores the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

12.10.5 Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

12.11 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 field samples.

12.11.1 Perform a level 1 data review using Data Review Checker in TALS and document the review in TALS.

12.11.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

12.12 Method Performance

12.13 Method Detection Limit Study (MDL)/Detection Limit

The method detection limit (MDL) or detection limit (DL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL/DL is determined according to the laboratory's MDL/DL procedure (see SOP TA-QA-0602). MDL/DLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL/DL studies for analyses performed; these are verified at least quarterly unless method requirements require a greater frequency.

For instruments that run samples which fall under LaMP regulations, a yearly MDL Study must be performed and MDLV starting at 1X the MDL.

12.14 Limit of Detection

The limit of detection (LOD) is determined for each analyte and matrix by spiking a quality system matrix at approximately two to four times the detection limit. This spike concentration establishes the LOD. The LOD is verified quarterly for each method and matrix on each instrument that analyzes said method/matrix. Refer to the laboratory's LOD procedure (see SOP TA-QA-0602)

12.15 Limit of Quantitation

The limit of Quantitation (LOQ) is verified quarterly for each method and matrix on each instrument that analyzed said method/matrix. Refer to the laboratory's LOQ procedure (see SOP TA-QA-0618)

12.16 Demonstration of Capabilities

Analyst initial Demonstrations of Capability (DOC) are performed after completing a read and understand memo for the SOP and before any client samples are analyzed. DOCs are updated annually (continuing DOC). See SOP TA-QA-0617 for details.

12.17 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Waste Disposal SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 VOA vials containing acidic water; VOA vials containing extracted acidic water and small amounts of methanol are collected in large plastic satellite waste bins marked "Hazardous Waste." At or before the waste reaches 55 gallons, the contents are transferred to the waste warehouse where the vials are bulked into a 55 gallon waste barrel and sent out for incineration.

14.1.2 VOA vials containing extracted soil samples, which will contain small amounts of methanol and possibly sodium bisulfate. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days have passed these sample extracts are transported to the waste room were they are bulked into the flammable liquid loose pack barrel and sent out for incineration.

14.1.3 Expired Standards. Expired standards are collected in satellite containers marked "Hazardous Waste." At or before the containers reach 55 gallons the containers are taken to the waste warehouse where they are bulked into an expired standards lab pack and sent out for incineration.

15.0 References / Cross-References

15.1 Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, August, 2006, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Fourth Edition, EPA Office of Solid Waste.

15.3 Method 5035A, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July, 2002, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.4 Department of Defense (DoD), Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories Based on ISO/IEC 17025:2005(E) and The NELAC Institute (TNI) Standards, Volume 1, (September 2009), DoD Quality Systems Manual Version 5.1 DOE Quality Systems for Analytical Services Version 3.1, 2017.

16.0 Method Modifications:

Item	Method	Modification
1	8260B	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
2	8260C	Storage conditions for standards are listed as <6C or as recommended by manufacturer. TestAmerica Seattle follows 8260B guidelines to store standards at -10C or as recommended by manufacturer.
3	8260C	The minimum RF values for 2-Hexanone are based on a lab study in full scan mode and not as listed in the method.
5	5035A	The aliquot of methanol extract taken for analysis is 125 uL rather than the 100 uL specified in Table 1 of the method.

17.0 Tables and Appendices

18.0 Revision History

- Revision 0, dated 3 May 2017
  - Initial release.

Table 1

TestAmerica Primary List Reporting Limits for 8260B SIM and 8260C SIM

Compound	CAS Number	Reporting Limits[1]	
		Water (µg/L)	Soil (µg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	0.5	5
1,1,2,2-Tetrachloroethane	79-34-5	0.5	10
1,1,2-Trichloroethane	79-00-5	0.5	5
1,1-Dichloroethene	75-35-4	0.5	5
1,2-Dichloroethane	107-06-2	0.5	5
1,4-Dichlorobenzene	106-46-7	0.5	5
2-Hexanone	591-78-6	0.5	30
Benzene	71-43-2	0.5	5
Bromoform	75-25-2	0.5	5
Bromomethane	74-83-9	0.5	5
Chlorodibromomethane	124-48-1	0.5	5
Chloroform	67-66-3	0.5	5
cis-1,2-Dichloroethene	156-59-2	0.5	5
cis-1,3-Dichloropropene	10061-01-5	0.5	5
Dibromomethane	74-95-3	0.5	5
Dichlorobromomethane	75-27-4	0.5	5
Ethylene Dibromide (EDB)	106-93-4	0.5	5
Hexachlorobutadiene	87-68-3	0.5	5
Naphthalene	91-20-3	0.5	5
Tetrachloroethene	127-18-4	0.5	5
trans-1,3-Dichloropropene	10061-02-6	0.5	5
Trichloroethene	79-01-6	0.5	5
Vinyl chloride	75-01-4	0.5	20

1 Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 2

8260B SIM and 8260C SIM Additional Analyte List Reporting Limits

Compound	CAS Number	Reporting Limits[1]	
		Standard Level Water µg/L	High Soil µg/kg
Butadiene	106-99-0	0.5	5
Isopropyl alcohol	67-63-0	2	70

1 Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 3

Typical Calibration Levels

Standard level water, Methanol preserved soil purge	Calibration Levels, µg/L							
	0.01	**0.05**	**0.1**	**0.5**	**1.0**	**5.0**	**10**	**25**

Table 4

Internal Standards

Internal Standard	Standard Concentration (mg/L)	Quantitation Ion
1,4-Dichlorobenzene-d4	25	152
Chlorobenzene-d5	25	117
Fluorobenzene	25	96
Tert-butanol-d9	500	65

Table 5

Surrogate Standards

Surrogate Compounds	Standard Concentration (mg/L)
1,2-Dichloroethane-d4	25
4-Bromofluorobenzene	25
Dibromofluoromethane	25
Toluene-d8	25
Trifluorotoluene	25

NOTES:

1) Recovery and precision limits for the surrogates are generated from historical data and are maintained by the QA department.

Table 6

Full List LCS and Matrix Spike Compounds

Compound	Standard Concentration (mg/L)
1,1,1,2-Tetrachloroethane	50
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethene	50
1,2-Dichloroethane	50
1,4-Dichlorobenzene	50
2-Hexanone	250
Benzene	50
Bromoform	50
Bromomethane	50
Butadiene	50
Chlorodibromomethane	50
Chloroform	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Dibromomethane	50
Dichlorobromomethane	50
Ethylene Dibromide (EDB)	50
Hexachlorobutadiene	50
Isopropyl alcohol	500
Naphthalene	50
Tetrachloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50
Vinyl chloride	50

NOTES:

1) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.

Table 7

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	50 to 120 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 8
Poorly Performing Compounds

2-Hexanone
Bromomethane
Bromoform
Naphthalene
Isopropyl alcohol

This is not a comprehensive list and is subject to change. Each DoD projects' target analyte list should be evaluated for poor performers.

Analytes that are in bold are also represented in Table 1 Reporting Limits for Standard Analytes.

Table 9

Summary of QC Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
BFB Tune	Prior to ICAL and at the beginning of each 12-hour period.	See Section 11.3	Retune instrument and verify. Rerun affected samples.
Minimum 5-point Initial Calibration, 6-point for quadratic curves (minimum 3-point Initial Calibration for Method 624)	Initial calibration prior to sample analysis	1. Average Response Factor for specified compounds: See table 10 2. RSD for all compounds: ≤ 20% **For DOD requirements above and one option below:** Option 1: RSD for each analyte ≤ 15% Option 2: Linear regression least squares regression r^2 ≥ 0.990 Option 3: Non linear least squares regression r^2 ≥ 0.990 and 6 points must be used.	Terminate analysis; correct the problem; recalibrate. Problem must be corrected. No samples may be run until ICAL has passed.
ICV or QCS	Following initial calibration.	70-130% for non-DoD projects and 45-155% for poor performers **For DoD:** 80 - 120% recovery and 70-130% for poor performers with prior written approval	Terminate analysis; correct the problem; recalibrate.
Relative Retention Times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL. Laboratory may update RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (e.g. column clipping).
CCV	Daily before sample analysis and every 12 hours of analysis time.	1. Avg RF for specified compounds: See table 10 2. %D/Drift for 80% of	Correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all sample

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		compounds ≤ 20%D. **For DoD:** 1. Avg RF: see method 2. Opening CCV: %D/Drift for all target compounds and surrogates ≤ 20%D. 3. Closing CCV: %D/Drift for all target compounds and surrogates ≤ 50%D.	since the last successful CCV.
Internal Standards (IS) verification	Every field sample, standard, and QC sample	Retention time ± 30 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard. **For DoD:** Retention time ± 10 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples while system was malfunctioning is mandatory.
Method Blank	One per batch of 20 field samples or fewer.	The result must be < RL or < 5% the amount measured in any sample or 1/10 the regulatory limit. **For DoD:** No analytes detected > ½ RL and > 10% the amount measured in any sample or 1/10 the regulatory limit. For common laboratory contaminants no analytes detected > RL.	Re-extract and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
LCS	One per batch of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported. QC acceptance criteria specified by DoD,	See Section 9.5 for additional requirements.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		if available. Otherwise, use in-house control limits.	
Surrogate	All field and QC samples.	Must be within laboratory control limits, unless it fails high and the sample is ND, or matrix interference is confirmed by a reanalysis or MS/MSD performed on the sample, or client specific requirements exist. **For DoD:** QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	See Section 9.4 for additional requirements.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported and must use LCS control limits. MD/MSD RPD ≤20%	See Section 9.5 for additional requirements.

Table 10

8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
1,1-Dichloroethene	0.100	0.313
cis-1,2-Dichloroethene	0.100	0.376
Chloroform	0.200	0.557
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.060	0.536
Bromoform	0.100	0.413
1,1,2,2-Tetrachloroethane	0.300	0.782
1,4-Dichlorobenzene	0.500	1.427

a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

b Data provided by EPA Region III laboratory.

Table 11
Characteristic Ions

Compound	Approx Retention Time (min)	Primary	Secondary	Tertiary
Internal Standards				
TBA-d9	6.716	65	46	--
Fluorobenzene	9.612	96	77	--
Chlorobenzene-d5	12.852	117	82	--
1,4 Dichlorobenzene-d4	14.864	152	150	115
Surrogates				
Dibromofluoromethane	6.586	113	111	--
1,2-Dichloroethane-d4	4.856	65	67	--
Trifuorotoluene	10.132	146	--	--
Toluene-d8	11.216	98	100	--
4-Bromofluorobenzene	13.701	174	95	176
Target Analytes				
Vinyl chloride	4.857	62	64	--
Butadiene	5.014	54	53	39
Bromomethane	1.679	94	96	--
1,1-Dichloroethene	6.786	96	61	98
Chloroform	8.478	83	85	--
Benzene	9.451	78	77	--
1,2-Dichloroethane	9.030	62	64	98
Trichloroethene	9.988	132	95	60
Dibromomethane	9.935	174	93	81
Dichlorobromomethane	10.024	83	85	47
cis-1,3-Dichloropropene	10.561	75	110	39
trans-1,3-Dichloropropene	10.948	75	110	49
1,1,2-Trichloroethane	11.097	97	83	61
2-Hexanone	11.395	58	43	85
Chlorodibromomethane	11.603	129	127	79
Ethylene Dibromide	11.841	107	109	81
Tetrachloroethene	11.990	164	131	166
1,1,1,2-Tetrachloroethane	12.535	131	133	117
Bromoform	13.110	173	175	171
1,1,2,2-Tetrachloroethane	13.313	83	85	131
1,4-Dichlorobenzene	14.888	146	111	75
Naphthalene	17.384	128	012	127
Hexachlorobutadiene	12.162	225	227	223

APPENDIX I: EXAMPLE SEQUENCE AND PROCESS FLOW

Example Instrument Sequence

GC/MS VOA ANALYSIS RUN LOG

Lab Name: TestAmerica Seattle Job No.: 580-46744-1

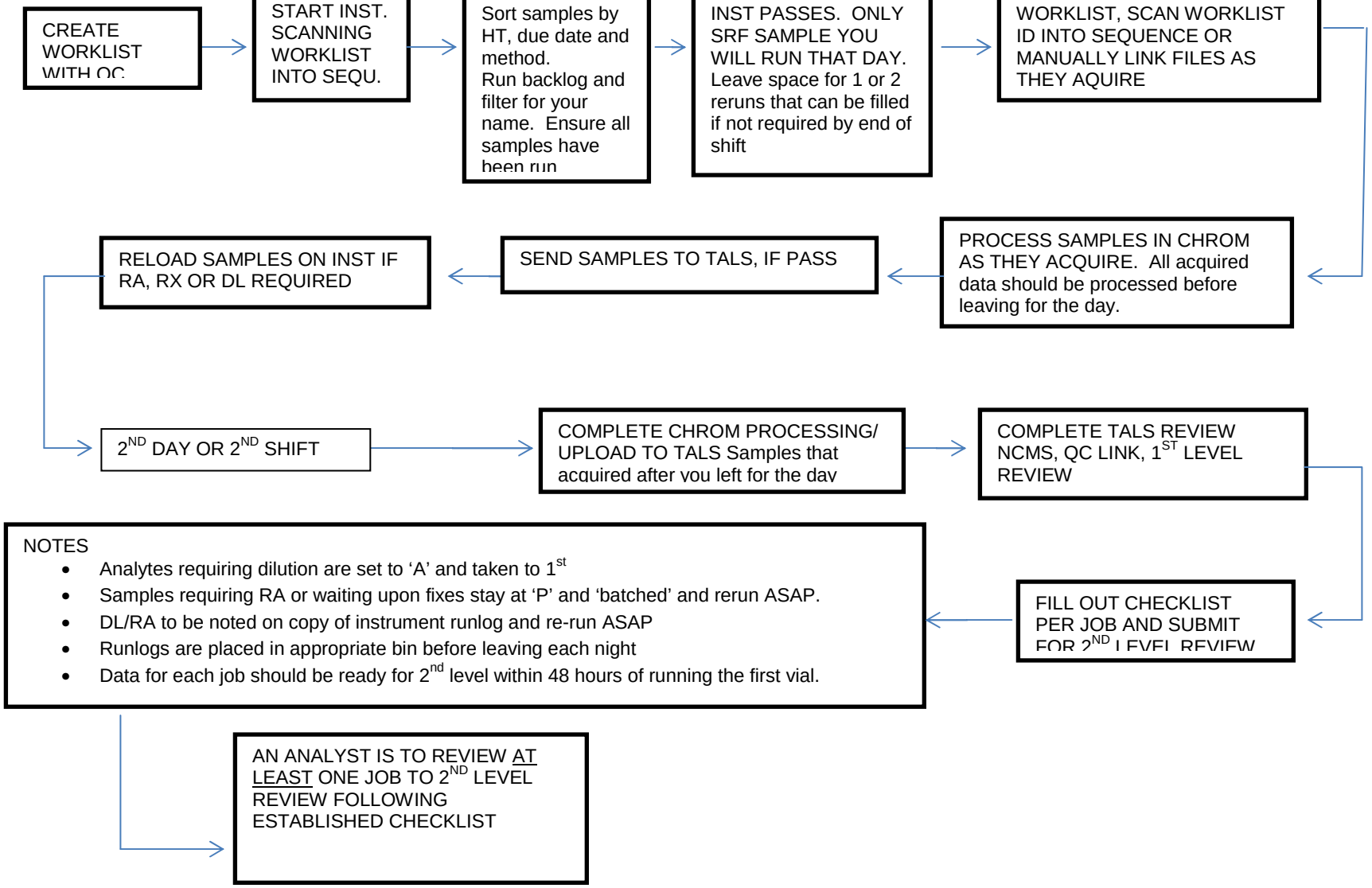
SDG No.:

Instrument ID: TAC041 Start Date: 12/06/2014 14:01

Analysis Batch Number: 178602 End Date: 12/06/2014 23:23

LAB SAMPLE ID	CLIENT SAMPLE ID	DATE ANALYZED	DILUTION FACTOR	LAB FILE ID	COLUMN ID
RTC 580-178602/2		12/06/2014 14:01	1		ZB-624 0.32(mm)
STD15000 580-178602/3 IC		12/06/2014 14:28	1	MS00187256.D	ZB-624 0.32(mm)
STD10000 580-178602/4 IC		12/06/2014 14:55	1	MS00187257.D	ZB-624 0.32(mm)
STD5000 580-178602/5 IC		12/06/2014 15:21	1	MS00187258.D	ZB-624 0.32(mm)
STD1000 580-178602/6 IC		12/06/2014 15:48	1	MS00187259.D	ZB-624 0.32(mm)
STD500 580-178602/7 IC		12/06/2014 16:15	1	MS00187260.D	ZB-624 0.32(mm)
STD250 580-178602/8 IC		12/06/2014 16:45	1	MS00187261.D	ZB-624 0.32(mm)
STD100 580-178602/9 IC		12/06/2014 18:14	1	MS00187262.D	ZB-624 0.32(mm)
STD50 580-178602/10 IC		12/06/2014 22:56	1	MS00187263.D	ZB-624 0.32(mm)
ICV 580-178602/11		12/06/2014 23:23	1		ZB-624 0.32(mm)

Appendix C; PROCESS FLOW FOR SEATTLE VOA



Appendix C – Site-Specific Health and Safety Plan (Under Separate Cover)

(This Page Intentionally Left Blank)

Appendix D – Field Forms

(This Page Intentionally Left Blank)

Daily Quality Assurance / Quality Control Report

QC/QA Report Number: 001

Day/Date: Month XX, 200X

Contract No: Name, Title

Contractor: Ahtna Environmental, Inc.

UPC Project Title: Name, Title

Location: Place

Weather

Low Temp:

High Temp:

Wind Speed:

Conditions:

Quality Control Inspections Performed This Date

(include inspections, results, deficiencies observed, and corrective action)

☐ Preparatory Phase (see attached checklist)

☐ Initial Phase (see attached checklist)

☐ Follow-Up Phase (see attached checklist)

Notes/Comments:

Field Sampling and Testing

Was sampling and testing performed this date? ☐ Yes ☐ No

Type of Test:

Method/Matrix:

Results:

Have Data Quality Objectives been achieved? ☐ Yes ☐ No ☐ N/A

Notes/Comments:

Laboratory Analytical Sampling

Was laboratory analysis sampling performed this date? ☐ Yes ☐ No

Type of Test:

Method/Matrix:

Quantity of Samples:

Have required amount of QC trip blanks been achieved? ☐ Yes ☐ No ☐ N/A

Have appropriate QC laboratory tests been ordered? ☐ Yes ☐ No ☐ N/A

Have QA and QC samples been collected in the specified quantity?

☐ Yes ☐ No ☐ N/A

Have samples been properly labeled and packaged? ☐ Yes ☐ No ☐ N/A

Notes/Comments:

Health and Safety

Worker protection levels this date (A, B, C, D, etc.):

Was any work activity conducted within a confined space? ☐ Yes ☐ No

Was any work activity conducted within an area determined to be IDLH?

☐ Yes ☐ No

Were approved decontamination procedures used on workers and equipment as required?

☐ Yes ☐ No ☐ N/A

Was a Job Safety Meeting held this day? ☐ Yes (see attached checklist) ☐ No

Were there any "Lost Time" accidents this day? ☐ Yes (see attached report) ☐ No

Was hazardous waste or materials released into the environment? ☐ Yes ☐ No

Notes/Comments:

Work Activities Performed This Date

(include location, description, and by whom. Also include operating equipment with hours worked, idle time, or down for repair)

Performed by Prime

Performed by Subcontractors

Labor and Equipment Used This Date

Contractor – Ahtna Environmental, Inc.

Labor			Equipment		
Classification	**Number**	**Hours**	**Type/Make**	**Number**	**Quantity/Unit**
Total					

Subcontractor –

Labor			Equipment		
Classification	**Number**	**Hours**	**Type/Make**	**Number**	**Quantity/Unit**
Total					

Subcontractor –

Labor			Equipment		
Classification	**Number**	**Hours**	**Type/Make**	**Number**	**Quantity/Unit**
Total					

Materials Received to be Used or Incorporated into Site

-
-

Equipment Received to be Used on Site

-
-

Instructions Given by the Government to the Contractor

☐ Verbal ☐ Written (include names, reactions, and remarks)

-
-

Instructions Given by the Contractor to Subcontractors

☐ Verbal ☐ Written (include names, reactions, and remarks)

-
-

Scope of Work / Work Plan Variances

Were there any variances this date? ☐ Yes ☐ No

-
-

Work Progress

Are there any contractor-caused delays?	☐ Yes	☐ No
Is there any contractor potential finding of fact?	☐ Yes	☐ No
Are there any government-caused delays?	☐ Yes	☐ No
Is there any government potential finding of fact?	☐ Yes	☐ No
Are there any unforeseeable or weather-related delays?	☐ Yes	☐ No

Notes/Comments:

Deficiencies Noted This Day

(include corrective actions taken and anticipated date of correction if carried over past COB)

Were there any deficiencies this date? ☐ Yes ☐ No

Deficiencies Corrected This Day

Were there any deficiencies corrected this date? ☐ Yes ☐ No

Regulatory Notifications This Day

Were there any regulatory notifications this date? ☐ Yes ☐ No

---

Remarks

(include visitors, etc.)

Contractor Verification Statement

The above report is complete and correct, and all work, workmanship, equipment, and materials received are believed, to the best of my knowledge, to be in compliance with State, Federal, and contract requirements.

_____ _____

CQC Systems Manager Signature Date

Site Superintendent Review and Comments:

_____ _____

Site Superintendent Signature Date

CALIBRATION LOG

Project: _____

Instrument Name _____

Mfg Model No. _____

Serial No. _____

Date	Time	Temperature	Calibration Standard Type	Lot No.	Exp. Date	Standard Concentration	Pre-Calibration Reading	Post-Calibration Reading

(This Page Intentionally Left Blank)

PROJECT PHOTOGRAPH LOG

Project:

Count	Photo ID	Date	Time	Taken By	Location	Description	Orientation
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

(This Page Intentionally Left Blank)

Ahtna Engineering Services, LLC	**GROUNDWATER SAMPLING FORM**	**PROJECT NUMBER:**	**WELL NUMBER:**	**SHEET:** of

PROJECT NAME		**WELL CONDITION**	
CLIENT		**DAMAGE PRESENT**	
DATE		**DEPTH TO BASE (FROM TOC)**	
SITE		**DEPTH TO WATER (FROM TOC)**	
GEOLOGIST		**HEIGHT OF WATER COLUMN**	
WEATHER/ TEMPERATURE		**WELL VOLUME**	
WIND		**3 WELL VOLUMES**	

NOMINAL DIAMETER	**O.D.**	**I.D.**	**VOLUME (GAL/LIN FT)**
1"	1.315"	1.049"	0.04
1.5"	1.9"	1.610"	0.11
2"	2.375"	2.067"	0.17
3"	3.5"	3.068"	0.38
4"	4.5"	4.026"	0.66

SAMPLING DATA

SAMPLE TYPE (GW, PRODUCT, OTHER):

SAMPLE COLLECTED WITH: __ Bailer ____ Pump, Type: _____ ___ Other, Specify:

MADE OF: __ Stainless Steel ____ PVC

__ Teflon ____ Disposable LDPE ___ Other, Specify:

SAMPLING DECON PROCEDURE:

SAMPLE DESCRIPTION: (color, free product thickness, odor, turbidity)

FIELD WATER QUALITY PARAMETERS

						Stabilization Requirements (3 must be stable)						
						3%	10%	0.1	10 mV	10%		
Time	**Purged Volume (Gal)**	**Purge Rate (mL/min)**	**Water Level**	**Draw Down (ft)**	**Temperature (°C)**	**Spec. Cond. (µS/cm)[c]**	**D.O. (mg/L)**	**pH**	**ORP (mV)**	**Turbidity (NTU)**	**Color**	**Odor**

ANALYTICAL SAMPLE INFORMATION

Sample ID	**Time**	**Analytes**
		DRO RRO GRO BTEX PAH VOCs PEST HERB
		DRO RRO GRO BTEX PAH VOCs PEST HERB
		DRO RRO GRO BTEX PAH VOCs PEST HERB

Sampling Notes:

(This Page Intentionally Left Blank)

FIELD CHANGE REQUEST (FCR)

CONTRACT TASK ORDER NAME:	**CTO #**	**CHANGE REQUEST NO. #**
TO:	**LOCATION:**	**DATE:**

RE:

_____ Specific Sections: _____ Title:_____

_____ Drawing # _____Title: _____

_____ Other: _____

1. DESCRIPTION (items involved, submit sketch, if applicable):

2. REASON FOR CHANGE

3. RECOMMENDED DISPOSITION (Submit sketch, if applicable):

_____ Minor Change _____ Major Change (Impacts Cost, Schedule)

4. DISPOSITION: (Approval Required by Client Representative)

_____ Considered minor change – APPROVED per recommended disposition – Documents will not be formally revised. Field office to maintain as –built records.

_____ Considered major change – Client approval required via contract modification process

_____ Not Approved (give reason).

Prepared by (Signature)	**Date:**
USACE Project Manager / OE Safety Specialist	**Date:**
Contractor Project Manager (Signature)	**Date:**
	Date:

(This Page Intentionally Left Blank)

Appendix E – ADEC Comment and Response

(This Page Intentionally Left Blank)

DEC Comments

Document:	NPS Indian River Asphalt Plant Preliminary Assessment/Site Inspection - Sampling and Analysis Plan
Date:	7/24/2018
Received:	7/13/2018
File No.:	1525.38.055

Comment No.	Page	Section	Comment / Recommendation	Response
1.	2-1	2.1	Please expand the background section to include additional details on the original facility including: 1. Were mixing tanks present on site to mix the asphalt? 2. What products were stored on site, how were they stored, and where were they stored? 3. Were any historical releases known to have occurred on site? 4. What structures existed at the site (i.e., USTs, ASTs, buildings, etc.)?	As discussed in our August 7, 2018 phone conference, the presence and location of these features are not known at this time, and no known record exists, as the facility was shut down in the late 1950's.
2.	1-2	1.2	DEC recommends adding GRO, DRO, and RRO into the list of COPCs to be evaluated, to align with the requirements of Appendix F in DEC's Field Sampling Guidance and prevent data gaps as the site moves into the RI phase.	These have been added as COPCs and are annotated in section 2.2.3.
3.	2-2	2.1.1	Was the estimated 400 cubic yards of contaminated soil estimated in the 1995 ESA what was removed from the site in 2017? Please clarify.	Our understanding is that only the asphalt mass was removed in 2017. We do not know if this was within the area of the 400 cubic yards estimated in the 1995 ESA.
4.	2-4	2.2.4	Please add soil as a potentially impacted medium.	Soil will be added to the impacted media.
5.	4-2	Table 4-1	Please discuss the exceedance of 2,4-Dinitrotoluene. Can insight be provided why this contaminant would be present on this site?	Per our discussion during the August 7, 2018 phone conference, this was a constituent of the asphalt; its exact purpose unknown.

6.	4-3	4.3.2	To better understand the impacted media, provide for the evaluation of risk at the site, and prevent data gaps as the site moves into the RI/Risk Assessment phase DEC recommends: 1. adding sediment samples to the SAP and collocating them to the pore water samples and 2. adding soil samples to the SAP. Section 2.2.2 indicates soil sampling from previous assessments will be used to close identified data gaps, however past soil samples were not collected under a DEC approved work plan and potentially do not represent current soil conditions as they were collected in 1995. The SPLP will aid in understanding the tar/asphalt product impacts to soils, however other releases, in addition to the tar/asphalt, cannot be ruled out for this type of operation and have potentially impacted soils.	Additional sampling will be addressed in a later phase of work for this site, as agreed upon in our August 7, 2018 phone conference.
7.	5-1	5.1	How will the location of pore water samples be chosen?	Exact locations will be based on accessibility and the professional judgement of the Ahtna Scientist on site. As stated in the current work plan, one pore sample location will be upgradient from the former asphalt mass, one will be located below the former asphalt mass, and the third will be located at the lower end of the property.
8.		Figure 2	Please indicate on the figure where the large mass was removed from, any other past excavations, location of past sampling efforts, where known contaminated materials left behind are located, and where erosion exposing buried materials is occurring. Also, please add a groundwater flow direction arrow if known.	The Asphalt mass will be added to the Site Plan Figure and included in the final SAP. Based on topography, we assume groundwater flow direction is southwest to

				southeast (and may locally vary with tidal flow), however it is not known.
9.		General	Please add an additional figure or an as-built that address Comment 1 above to identify original site features including: facility layout, USTs or ASTs, mixing tanks, structures, product storage, etc.	See response to comment 1.
10.		General	If possible, please provide to DEC the documentation or reporting of contaminant related activities at the site (e.g., 1995 Site Assessment, 2002-2005 water quality monitoring).	This was provided to ADEC by NPS on July 27, 2018.

(This Page Intentionally Left Blank)

Appendix F – ADEC Comment and Response

(This Page Intentionally Left Blank)

THE STATE of ALASKA
GOVERNOR MICHAEL J. DUNLEAVY

Department of Environmental Conservation
SPILL PREVENTION & RESPONSE
Contaminated Sites Program

610 University Avenue
Fairbanks, Alaska 99709
Main: 907.451.2143
Fax: 907.451.2155
www.dec.alaska.gov

File: 1525.38.055

March 4, 2019

Bill Heubner
National Park Service
240 West 5th Avenue
Anchorage, AK 99501

RE: DEC Approval - NPS Preliminary Assessment and Site Inspection Report for Sitka National Historical Park Indian River Asphalt Plant

Dear Mr. Heubner:

Thank you for providing the NPS Preliminary Assessment and Site Inspection Report for Sitka National Historical Park, Indian River Asphalt Plant to Alaska Department of Environmental Conservation (DEC). This report describes the pore water sampling and asphalt leachate analysis in the area of remaining petroleum contamination from a historic asphalt operation. Pore water sampling indicates remaining petroleum contamination is not directly impacting surface waters. Leachate from the asphalt mass remaining indicates precipitation water leaching should not result in the migration of contamination from the asphalt.

DEC has reviewed the report and it is approved with the following comments. DEC concurs with the recommendations for the re-investigation of soils in the following historic test pit locations: TP-6, TP-9, and TP-10, however, in addition DEC recommends that the location of lead exceedances in TP-7 also be re-investigated. DEC also concurs groundwater impacts should be investigated during the next field effort.

Please provide the final version of the Preliminary Assessment when prepared. If you have any questions or concerns please contact me at (907) 451-2370 or gretchen.caudill@alaska.gov.

Sincerely,

G. Caudill

Digitally signed by Gretchen Caudill
DN: cn=Gretchen Caudill, o=DEC, ou, email=gretchen.caudill@alaska.gov, c=US
Date: 2019.03.04 15:47:57 -09'00'

Gretchen Caudill
Environmental Program Specialist

cc: Nino Muniz, Ahtna
Alexa Fitzgerald, Ahtna

(This Page Intentionally Left Blank)