Draft
Remedial Investigation/Feasibility Study Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

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Prepared for

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LIST OF ABBREVIATIONS AND ACRONYMS

µg/L	micrograms per liter
µg/m^3	micrograms per cubic meter
ADCS	Air Defense Command System
Airport	Snohomish County Airport
ARAR	applicable or relevant and appropriate requirement
AST	aboveground storage tank
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, xylenes
CDM	Camp Dresser & McKee Inc.
CFR	Code of Federal Regulations
CLARC	Cleanup Levels and Risk Calculations
County	Snohomish County
CUL	cleanup level
DAHP	Washington State Department of Archaeology and Historic Preservation
DCA	dichloroethane
DCE	dichloroethene
DCP	dichloropropane
DRO	diesel-range organics
DRPH	diesel-range petroleum hydrocarbons
Ecology	Washington State Department of Ecology
EPA	US Environmental Protection Agency
ESA	environmental site assessment
FS	feasibility study
ft	foot/feet
ft^2	square foot/feet
Giddens Trust	Thomas V. Giddens Living Trust
GPR	ground-penetrating radar
GRO	gasoline-range organics
HASP	health and safety plan
HRPH	heavy-range petroleum hydrocarbons
LAI	Landau Associates, Inc.
MCL	maximum contaminant level
mg/kg	milligrams per kilogram
MTCA	Model Toxics Control Act
ORO	oil-range organics
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCOC	potential contaminant of concern

LIST OF ABBREVIATIONS AND ACRONYMS (CONTINUED)

QAPP	quality assurance project plan
RAO	remedial action objective
RCW	Revised Code of Washington
RI	remedial investigation
SAP	sampling and analysis plan
TCA	trichloroethane
TCE	trichloroethene
TECT	TECT Aerospace
TOC	total organic carbon
TPH	total petroleum hydrocarbons
TPH-D	diesel-range total petroleum hydrocarbons
TSCA	Toxic Substances Control Act
UNC	United Nuclear Corporation
USACE	US Army Corps of Engineers
UST	underground storage tank
VOC	volatile organic compound
WAC	Washington Administrative Code

1.0 INTRODUCTION

This document presents a work plan to conduct a remedial investigation (RI) on Snohomish County Airport (Airport) property currently and formerly leased to TECT Aerospace (TECT) and on the former East Fuel Farm property. These areas, collectively referred to as the Site, are located in the southeastern portion of Sector 5 of the Airport in Everett, Washington (Figure 1). The Site includes existing Buildings C-19, C-20, C-21, C-22, C-23, as well as former Building C-29 and the former East Fuel Farm (Figure 2).

This work plan was prepared by Landau Associates, Inc. (LAI) on behalf of Snohomish County (County) and was developed to meet the general requirements for an RI as defined by the Washington State Department of Ecology (Ecology) Model Toxics Control Act (MTCA) cleanup regulation (Washington Administrative Code [WAC] 173-340-350). This work plan describes the RI activities to be performed and the planned schedule for data collection, evaluation, and reporting and the procedures to be used for completion of a Feasibility Study (FS). This work plan also includes a sampling and analysis plan (SAP; Appendix A), a quality assurance project plan (QAPP; Appendix B), and a Health and Safety Plan (HASP; Appendix C) for the RI.

1.1 Investigation Objectives Overview

A number of environmental investigations and environmental property assessments have taken place at the Site since the 1990s. Pre-RI exploration locations are shown on Figure 3. These investigations have identified subsurface contamination at the Site including the following:

- Chlorinated solvents in shallow perched groundwater and in the deep Esperance Sand aquifer
- Chlorinated solvents in soil and soil gas
- Petroleum hydrocarbons and metals in shallow soil and groundwater.

The previous investigations were generally focused on a specific building or buildings, or other specific features (e.g., former East Fuel Farm) of the Site. A comprehensive investigation of environmental conditions at the Site has not been completed.

The County plans to redevelop a major portion of the Site and wishes to address environmental contamination issues at the property prior to redevelopment. The RI will investigate the subsurface soil and groundwater at the Site and characterize the concentration, chemical nature, extent (horizontal and vertical), and the direction and rate of migration of contaminants of concern released into the environment at or from each of the investigation areas identified during previous investigations. The investigation areas for the RI are grouped by building usage and shown on Figure 4. In addition, the RI will evaluate potential human and environmental receptors and potential pathways to each receptor from each investigation area, as appropriate, and support recommendations for potential interim measure(s) or an FS of remedial action alternatives, if warranted.

A desired outcome of the RI/FS will be the development and implementation of a cleanup action plan that leads to a No Further Action (NFA) determination by Ecology for the Site; however, an NFA determination may not be required provided the cleanup is compatible with Site redevelopment plans.

1.2 Investigation Overview

The RI will include focused investigation of soil, groundwater, and soil gas associated with the following areas at the Site that were identified during previous investigations:

- Building C-19
- Building C-20, C-21, C-22 Complex
- Building C-23 and C-23 Annex
- Former Building C-29 / Former East Fuel Farm
- Deep Aquifer.

The RI will likely be implemented in phases. The sequence of activities is expected to be as follows:

1. Utility investigation work and collection and analysis of groundwater samples from existing monitoring wells
2. Collection and analysis of soil, groundwater, and/or soil gas grab samples from each of the investigation areas (to be conducted during winter months when shallow groundwater is most likely to be present)
3. Installation of shallow and deep monitoring wells at selected locations throughout the Site to allow for the sampling and analysis of groundwater and to monitor seasonal groundwater flow direction and gradient, and forensic analysis for petroleum hydrocarbons of selected soil, groundwater, and petroleum product samples.
4. Installation and sampling of a second round of deep monitoring wells, if warranted, based on the findings from the earlier phases of investigation.

Preliminary locations for the grab samples, shallow monitoring wells, and the first phase of deep monitoring wells are identified in this work plan. The monitoring well locations, and/or the number of wells, may be adjusted based on the results of initial phases of investigation. The final locations for the monitoring wells will be discussed and agreed to with the County prior to installation, along with any other potential follow-on actions that may be warranted based on review of the data collected during the initial phases of investigation.

2.0 SITE DESCRIPTION AND BACKGROUND

This section describes features of the Site, current and future land and water use, and historical Site uses.

2.1 Current Site Features

The Site is approximately 10 acres in size and is located in the southeastern portion of the Airport, as shown on Figure 1. The location of the Site and the approximate Site boundary is shown on Figure 2. The Site is generally bounded by Runway 34-16 and associated taxiways on the east, a paved surface parking lot followed by Paine Field Road to the south, 109th Street and 30th Avenue South to the west, and the Aviation Technical Services (ATS) Hangar 1 lease area to the north. The former East Fuel Farm is located within the boundary of the ATS lease area.

The Site is at an average elevation of approximately 600 feet (ft) above mean sea level. Surface topography at and in the vicinity of the Airport is generally flat. To the west of the Airport, the land surface slopes downward to the west toward Puget Sound, located approximately 2.5 miles west of the Site.

Predominant Site features are described as follows:

- The former East Fuel Farm is located at the north end of the Site. Underground storage tanks (USTs) remain in place; however, surface evidence of the USTs was not observed. This area currently consists of undeveloped land covered with mown grass, small trees, and blackberry plants.
- Building C-29 was previously located south of the former East Fuel Farm. The former building area is currently occupied by overgrown vegetation and is partially enclosed by a chain-link fence.
- Building C-23 occupies the north-central portion of the Site and consists of three sections: a 43,164-square-foot (ft^2) portion; a 2,400-ft^2 structure on the east side, containing the oil shed; and the 16,000-ft^2 Annex. Building C-23 is currently occupied by TECT and used for manufacture of aerospace parts. Grassy fields are located to the north and east of Building C-23.
- Buildings C-20, C-21, and C-22 are connected to each other by enclosed breezeways to form a large complex of buildings located to the south of Building C-23. These buildings are currently unoccupied. Until late 2017, these buildings were occupied by TECT and used for manufacture of aerospace parts.
- Building C-19 occupies the southern portion of the Site. Building C-19 is owned by the Thomas V. Giddens Living Trust (Giddens Trust), on land leased from the County. The building is currently occupied by Vector Industries, Inc., a metal fabrication company, and a sign company.

Much of the Site is paved, with some unpaved areas to the northwest, north, and east.

2.2 Current and Future Land Use

The Site is currently zoned for light industrial use (Snohomish County; accessed July 17, 2018). The Site land and buildings are owned by the County and leased to tenants for aerospace manufacturing and other light industrial operations, with the exception of Building C-19, which is owned by the Giddens Trust. The former locations of the East Fuel Farm and Building C-29 are currently unoccupied. Future land use is anticipated to be light industrial operations, and/or commercial activities, consistent with zoning and the current Site use.

2.3 Current and Future Groundwater Use

Groundwater at or potentially affected by the Site is not currently used for drinking water and future use of groundwater at the Site as drinking water is not anticipated. The drinking water source for Paine Field is Spada Lake, located approximately 8 miles north of Sultan, Washington. Drinking water at the Site is distributed by Mukilteo Water and Wastewater District and is treated by the City of Everett.

2.4 Site History

This section presents a brief history of the Airport and Site.

2.4.1 Snohomish County Airport History

The Airport was first developed in the late 1930s on a 640-acre parcel that was previously owned by the Puget Mill Company. Construction on the Airport began in 1936 and the Airport was operating 2 years later. The US Army Air Corps leased the Airport from the County in 1940 and acquired the Airport in 1941 for use as an Army airfield, named Paine Field. Between 1941 and 1942, the current Airport configuration was established. Military use of the airfield continued until October 1945 and the County regained ownership in 1948. In 1950, the US Air Force rehabilitated the old Army Air Corps facilities and improved runways as part of the Air Defense Command System (ADCS). From 1951 through 1968, the Airport served both the US Air Force (southern portion) and the County (northern portion). Construction of a BOMARC (long-range anti-aircraft) missile site began in 1957, to the northeast of the Site, but was not completed. In 1968, the US Air Force deactivated the ADCS portion of the Airport (LAI 1993).

2.4.2 Building C-19

The area of Building C-19 was historically developed with barracks associated with the former military operations at the Site. Building C-19 was constructed in 1979, and is owned by the Giddens Trust. The land is owned by the County and is under a long-term lease to the Giddens Trust. Since its construction, Building C-19 was operated along with Buildings C-20, C-21, C-22, and C-23 for aerospace parts manufacturing (see Section 2.4.3). In approximately 2013 or 2014, TECT Aerospace vacated Building C-19 and the building is currently occupied by Vector Industries, Inc., a metal fabrication company, and a sign company (Neil 2017).

2.4.3 Buildings C-20, C-21, C-22, and C-23

The Building C-20, C-21, C-22 complex was constructed in stages beginning in the 1950s. The southern portion of Building C-23 was originally constructed between 1941 and 1952 as two buildings, later connected by a breezeway. The east side of the building (oil shed) was added in 1978. The Annex was built on the north side of the building in 1987. This portion of the Site has been used for aerospace manufacturing since the 1950s. General operations have reportedly remained relatively unchanged, although the business names and ownership have changed over time. Previous operators of the facility have included Castle Industries (1950s), All Fab (1960s-1980s), Certified Holdings Corporation, United Nuclear Corporation (UNC), Grenwich, General Electric, Neuvant Aerospace, Prudential, and TECT (2004 to present). TECT consolidated its operations in Building C-23 in late 2017 and the Building C-20, C-21, C-22 complex is currently unoccupied.

Manufacturing processes/structures in this area of the Site include: maintenance shop, freezer, brake press, tumbler (Building C-20); sheet metal fabrication, forming, welding, heat treating (Building C-21); machining, stretch form, and hammer shop (Building C-22); and machining, hand forming, trimming, assembly, painting, and deburring (Building C-23).

2.4.4 Former Building C-29

Former Building C-29 (also referred to as the Meiers Building) was a chemical storage shed that was located west of the northwest corner of Building C-23 (Figure 2). The building is first visible in aerial photographs from 1947. The building was reportedly partially occupied by Meiers Wrought Iron from 1965 to 1981 and also by All Fab, Inc. The building was removed in January 1996 while being leased by UNC Aerostructures (Envirotech 1997). Contamination was discovered beneath the building at the time of demolition, as discussed in Section 3.3.

2.4.5 Former East Fuel Farm

The former East Fuel Farm is located northwest of Building C-23, and north of former Building C-29, as shown on Figure 2. The fuel farm was developed in the 1940s and was used by the US Army Air Corps during World War II and was later used by Alaska Airlines and Revolution Airlines for aviation fuel. At the time of an assessment completed in 1993, the fuel farm was operated by Flightline Services and used to supply aviation fuel for general aviation, military, and commercial aircraft (AGI 1993).

Records regarding the number, size, contents, and locations of USTs associated with the fuel farm are inconsistent. Based on a review of available records, the following USTs are or were present at the East Fuel Farm:

ID	Size (Gallons)	Contents	Status	Notes
93	25,000	Jet-A fuel	In place – temporarily abandoned in place in 1996	Upgraded in 1967 to store Jet-A fuel
95B	12,000	Aviation fuel	Removed in 1992	100 cubic yards of impacted soil removed at time of UST removal
Not Registered	Est. 12,000	Unknown	In place – identified during geophysical survey in 1994	
Not Registered	Est. 5,000-10,000	Unknown	Possible UST identified during geophysical survey in 1994	

Available records indicate that up to three USTs remain in place at the former East Fuel Farm. The tanks are not currently in use and have reportedly had their contents removed and been rinsed clean. Based on subsurface investigations completed at the former East Fuel Farm, as discussed in Section 3.4, subsurface contamination by petroleum hydrocarbons has been identified in this area.

3.0 PREVIOUS INVESTIGATIONS AND CLEANUP ACTIONS

Various environmental investigations and remedial actions have been conducted at the Site to characterize and evaluate the chemical quality and physical condition of soil, groundwater, and soil gas, or to address specific releases. This section briefly describes the environmental investigations and remedial actions previously conducted. The reader is referred to previous investigation reports for more detailed descriptions of the various previous investigations and remedial actions. Historical analytical data are provided in Tables 1 through 11.

The investigations described in the following sections were completed between 1992 and 2017 and data from these previous investigations are discussed in terms of MTCA cleanup levels (CULs) as reported at the time of the investigation. Data have also been evaluated relative to screening levels developed for the RI (see Section 5.0 for discussion on development of RI screening levels). Results of this evaluation are included in Section 6.0.

3.1 Building C-19

This section includes relevant findings from Phase I environmental site assessments (ESAs) and subsequent investigations at Building C-19. Previous investigations at Building C-19 have focused primarily on a former degreaser located at the south end of the building. The degreaser, 1,1,1-trichloroethane (1,1,1-TCA) storage tank, and a chiller were removed from the building in 1993 (LAI 1993). In addition, the building owner conducted an investigation and cleanup of petroleum-contaminated soil resulting from releases of cutting oil from machinery located in the manufacturing area of the building. Additional details are provided in the following sections and previous sampling locations are shown on Figure 5a.

3.1.1 1993 Phase I Environmental Site Assessment

A Phase I ESA was completed at Buildings C-19, C-20, C-21, C-22, C-23, and C-29 by LAI on behalf of Snohomish County in 1993. Building C-19 was occupied by All Fab, Inc. at the time of the assessment. Relevant finding from the Phase I ESA for Building C-19 are as follows (LAI 1993):

- Manufacturing operation areas at Building C-19 included a metal fabricating area, a sandblasting room, a former 1,1,1-TCA storage tank, dip tank (degreaser) and chiller area, oil drum storage areas and a sump, an acid dip tank room, and waste metal storage bins.
- Water-soluble cutting fluids were used throughout the manufacturing areas of the building. The site representative reported that chlorinated cutting fluids had been used in the past.
- A former degreaser pit was identified at the southern corner of the building. The pit was described as a 15-ft-wide by 40-ft-long by 8-ft-deep concrete-lined vault. A sump was located in the southeast corner of the vault and was used to collect spilled 1,1,1-TCA and groundwater that reportedly seeped into the sump. The sump was pumped as needed and the liquid was stored in drums prior to transport off site for disposal. The 1,1,1-TCA tank was reportedly situated on a concrete pad outside the building. A representative of All Fab reported that trichloroethene (TCE) and possibly other chlorinated solvents were used in the past.

- Severe oil staining was observed in the area of a waste oil drum on the southeast side of the building. The drum was used to collect oil drippings from scrap metal prior to placing the metal in an adjacent roll off bin.

The Phase I ESA concluded that there was potential discharges of 1,1,1-TCA and other solvents from the vapor degreaser to the local stormwater sewer system, natural features (such as Big Gulch), and soil and groundwater. In addition, the Phase I ESA concluded that there was potential for cutting fluids, including chlorinated cutting fluids, to penetrate the concrete floor of the building resulting in impacts to soil and groundwater.

3.1.2 Degreaser Sump Area

In 1994, on behalf of the Snohomish County Airport, LAI conducted a preliminary investigation of the vapor degreaser pit and sump in the southern corner of Building C-19 by excavating two test pits below the base of the concrete floor of the vapor degreaser pit (identified as C19-TP1 and C19-TP2 on Figure 5a). TCE was detected at concentrations above MTCA Method A CULs in effect at that time in soil and TCE, 1,1,1-TCA, 1,1-dichloroethane (1,1-DCA) and vinyl chloride were detected at concentrations above MTCA Method A or B CULs in groundwater. Additional volatile organic compounds (VOCs) were detected at concentrations below applicable CULs. Soil samples were collected at a maximum depth of 1.8 ft below the top of the concrete floor of the sump, and water samples were collected from perched water that accumulated in the test pits over several hours by seeping through the sides of the test pits and into the degreaser sump through weep holes in the concrete walls (LAI 1994).

In 1995, LAI conducted additional sampling and analysis in and around the degreaser sump, on behalf of Jack Giddens. Prior to sampling, the concrete floor of the sump and approximately 1 ft of underlying soil had been removed. Thirteen soil samples were collected from approximately 0.5, 1, and 2.5 ft below the base of the former sump floor and from approximately 0.5, 1, and 2 ft laterally beyond the former sump walls. Three water samples were collected from water seeping into the holes dug for collection of soil samples. The samples are identified by the prefix SU or SU2 on Figure 5a. TCE and vinyl chloride were detected at concentrations above MTCA Method A or B CULs in soil. TCE, 1,1-dichloroethene (1,1-DCE), and cis-1,2-DCE were detected at concentrations above MTCA Method A or B CULs in water (LAI 1995).

In 1999, on behalf of Snohomish County Public Works Department, AGI conducted a preliminary contamination assessment at Buildings C-19 and C-29 (see Section 3.3 for discussion of previous investigations at Building C-29). AGI identified and collected groundwater samples from three monitoring wells near Building C-19 that were installed by the Snohomish County Public Works Department in 1996 (SCPWD-2 through -4). Well depths ranged from 18 to 29.3 ft below ground surface (bgs) and depths to groundwater during sampling ranged from 2.47 ft bgs to 6.8 ft bgs. TCE was detected in groundwater at a maximum concentration of 140,000 micrograms per liter (µg/L) and detected concentrations exceeded the MTCA Method A CUL (5 µg/L) in water samples from all three wells. The concentration of tetrachloroethene exceeded the MTCA Method A CUL (5 µg/L) in water from SCPWD-3, and vinyl chloride concentrations exceeded the MTCA Method A CUL (0.2 µg/L) in water from SCPWD-2 and 3 (AGI 1999).

Camp Dresser & McKee Inc. (CDM) conducted a deep aquifer investigation in 2000 (discussed in more detail in Section 3.5), during which monitoring well DW1 was installed southwest of Building C-19. Aquifer groundwater was encountered at approximately 133 ft bgs and sampling and analysis of samples from DW-1 indicated that chlorinated VOCs had reached the deep aquifer (CDM 2000a).

In 2005, CDM reported the results of an investigation conducted for the Snohomish County Airport to delineate the lateral extent of chlorinated VOC contamination in shallow soil and perched groundwater near Building C-19. The investigation included the installation of 20 direct-push probes identified as GP1 through GP20 on Figure 3. CDM reported that TCE was not detected in soils at depths shallower than 5 ft bgs, which would have indicated a surface release. The core of the plume appeared to be located below 29th Avenue West (southwest of Building C-19) and appeared to extend in a west to east (downgradient) direction, migrating along sewer and storm drain lines. No offsite TCE source (other than Building C-19) was identified (CDM 2005).

3.1.3 Petroleum-Contaminated Soil beneath the Floor of Building C-19

Environmental Partners Inc. (EPI) completed site characterization at Building C-19 in 2005, 2006 and 2007 for the Giddens Trust. In 2005 and 2006, soil and shallow groundwater samples collected from beneath the floor of the interior of Building C-19 were analyzed for diesel- and heavy-range petroleum hydrocarbons (DRPH and HRPH) to evaluate potential subsurface impacts resulting from releases of cutting oil. Selected soil samples were also analyzed for polycyclic aromatic hydrocarbons (PAHs). DRPH and HRPH were detected at concentrations above the MTCA Method A CUL in shallow soil beneath the southern and central section of Building C-19. Neither DRPH nor HRPH was detected at a concentration above reporting limits in groundwater samples (EPI 2006, 2007). PAHs were detected in two soil samples at concentrations greater than the MTCA Method B cleanup level; the exceedances were co-located with exceedances for total petroleum hydrocarbons (TPH). In 2010, EPI conducted additional soil sampling and analysis in an area not accessible during previous investigations, and detected additional areas with DRPH and HRPH at concentrations above MTCA Method A CULs in soil beneath the northern section of Building C-19 (EPI 2010). During the site characterization sampling completed between 2005 and 2010, a total of 121 soil samples were collected from 65 locations throughout the manufacturing areas of the building. In addition, three shallow groundwater samples were collected from beneath the south end of the building and one shallow groundwater sample was collected from outside the east corner of the central portion of the building. The screened intervals for the temporary wells ranged from 5 to 13 ft bgs. The characterization sampling locations are not shown on the figures included with this document.

In 2011 and 2012, EPI reported the results of remedial actions completed in Bays 1, 2 and 3 (northern, central, and southern areas of Building C-19) conducted on behalf of the Giddens Trust. Shallow soil was excavated from six areas beneath the floor of Building C-19 and transported to a treatment facility. The locations of the excavation areas are shown on Figure 5a. Confirmation soil samples (a total of 88) were collected from the bottom and sidewalls of the excavations and analyzed for DRPH and HRPH. Detected concentrations were less than MTCA Method A soil cleanup levels in all samples. EPI requested an NFA determination from Ecology based on the remedial actions conducted in Bays 1, 2, and 3 (EPI 2011, 2012). Ecology issued an NFA determination for Bays 1, 2, and 3, as described above, but noted that the NFA determination did not apply to the TCE contamination near the degreaser sump, described above (Ecology 2013).

The historical investigation data described above were considered in developing the planned RI investigation locations discussed in Section 6.0 and shown on Figure 5d.

3.2 Buildings C-20, C-21, C-22, and C-23

This section includes relevant findings from previous Phase I ESAs and subsequent investigations at Buildings C-20, C-21, C-22, and C-23.

3.2.1 Phase I Environmental Site Assessment Findings

A Phase I ESA was completed at Buildings C-19, C-20, C-21, C-22, C-23, and C-29 by LAI on behalf of Snohomish County in 1993. The Building C-20, C-21, C-22 complex was referred to collectively as Building C-22 in the 1993 report and the buildings were occupied by All Fab, Inc. at the time of the 1993 assessment. A Phase I ESA was conducted by LAI on behalf of the County at Buildings C-20, C-21, C-22, and C-23 in 2017. The buildings were occupied by TECT at the time of the 2017 assessment. Relevant finding from the Phase I ESAs for Buildings C-20, C-21, and C-22 are as follows (LAI 1993, 2017):

- Manufacturing operations areas and other features observed in the Building C-20, C-21, C-22 complex in 1993 included metal fabricating areas (C-21), molten lead and alloy tanks (C-22), metal stretching machine and associated sump (C-22), a heat treating and glycol quench tank area (C-21), and a drop hammer machine area (C-22).
- The metal stretching machine was located in a rectangular vault in the southwest corner of Building C-22. The vault was observed to be approximately 6 ft deep and included a sump near the center of the vault. The sump reportedly collected cutting oil from the machine as well as groundwater that would seep into the vault. The sump was reportedly pumped as needed and the liquid was taken to an oil recycling area at Building C-23. The machine had been removed and the vault cleaned and filled by the time of the 2017 Phase I ESA.
- Two USTs were reportedly removed from the north side of Building C-22 in 1989. Minor soil contamination was reportedly encountered and excavated at the time of the UST removal; however, documentation was not available for review.

- A drainage trench was observed in Building C-22 and continuing west along the north side of the exterior of Building C-20. The 1993 report indicates that the trench drain is connected to the storm drainage system and to a detention pond located east of the Site. At the time of the 2017 Phase I ESA, the discharge point of the trench drain could not be determined.

At the time of the 2017 Phase I ESA, most manufacturing operations had ceased in the Building C-20, C-21, C-22 complex. Degraded and stained asphalt and concrete were observed in the manufacturing areas and storage drums and other containers of hazardous materials and petroleum products were observed without adequate secondary containment. The 2017 Phase I ESA concluded that there is a high potential for subsurface soil contamination to be present in areas where cutting oils and other hazardous materials and petroleum products were used and stored.

Relevant finding from the Phase I ESAs for Building C-23 are as follows (LAI 1993, 2017):

- Manufacturing operation areas at Building C-23 included metal fabricating areas, a waste storage area, chemical storage areas, and an empty drum storage and metal chip processing area.
- At the time of the 1993 assessment, a liquid waste storage area was identified on the west side of the building. The storage area included an approximately 2,000-gallon waste antifreeze tank, a 300-gallon evaporator, and a 500-gallon sludge tank. The tanks were located on a paved area within a berm. At the time of the 2017 assessment, a 3,000-gallon aboveground storage tank (AST) containing used coolant was located in this bermed area as well as a smaller AST (former evaporator) that is used for backup used coolant storage, if necessary. Staining and degraded concrete were observed in the vicinity of the ASTs.
- An oil shed was observed to the east of the center of Building C-23. This area was identified as a wood shop during the 1993 assessment. A sump is located in a covered area between Building C-23 and the oil shed. Metal chips resulting from the fabrication process were placed in chip bins and allowed to drain in this area. The sump is reportedly lined with concrete and has a solid bottom; however, the lining was not inspected during the 1993 or 2017 Phase I ESAs and staining and degraded concrete were observed.
- At least one out-of-use heating oil UST is located at the southwest corner of Building C-23. Previous reports include conflicting information regarding the number of USTs and a second UST is potentially present. The heating oil UST was reportedly emptied, but has not been filled, decommissioned, or removed.

Both the 1993 and 2017 Phase I ESA concluded that there is potential for subsurface contamination at Building C-23, resulting from current and historical industrial operations.

3.2.2 2017 Phase II Soil Investigation Results

In 2017, LAI conducted a focused Phase II investigation at the current TECT lease area (Buildings C-20, C-21, C-22, and C-23 and associated land) on behalf of the County. Additional details are provided in this and the following sections and historical sampling locations are shown on Figures 6a and 7a.

A total of 22 soil samples were collected from the TECT lease area and submitted to ALS Laboratory Group (ALS), of Everett, Washington for selected analysis for diesel-range and oil-range total petroleum hydrocarbons (TPH-D and TPH-O), VOCs, and glycols. The soil samples were collected using a direct-push probe rig or hand tools and are identified on Figure 3 as LAI-01 through LAI-28. The analytical results and exceedances of MTCA Method A CULs for industrial land uses are summarized below.

- Glycols were not detected at concentrations greater than the laboratory reporting limits in the one sample submitted for analysis.
- TPH-D or TPH-O was detected in 9 of 21 samples at concentrations greater than the laboratory reporting limits. The detected concentrations of TPH-D were less than the Method A CUL of 2,000 milligrams per kilogram (mg/kg). TPH-O was detected at concentrations greater than the Method A CUL at two locations:
    - TPH-O was detected at a concentration of 4,200 mg/kg at LAI-10 at a depth of 1 ft bgs. This sample was collected from along the trench drain located north of Building C-20.
    - TPH-O was detected at a concentration of 6,900 mg/kg at LAI-16 at a depth of 2.1 ft bgs. The sample was collected from beneath the floor of the machine shop located in the southwestern portion of Building C-23.
- The VOCs cis-1,2-dichloroethene (cis-1,2-DCE) and trichloroethene (TCE) were each detected in 4 of 17 samples at concentrations greater than the laboratory reporting limits. No other VOCs were detected in the samples.

  TCE was detected at concentrations greater than the Method A CUL of 0.03 mg/kg in four samples collected from three locations:
    - TCE was detected at a concentration of 4 mg/kg in the sample from LAI-25 at a depth of 15 ft bgs. The sample was collected from outside the southwest corner of Building C-22.
    - TCE was detected at concentrations of 0.04 mg/kg at a depth of 6.5 ft bgs, and 1.4 mg/kg at a depth of 9.5 ft bgs in samples collected from LAI-26. Sampling location LAI-26 was located outside the southwest corner of Building C-22.
    - TCE was detected at a concentration of 3.8 mg/kg in the sample from LAI-27 at a depth of 8 ft bgs. The sample was collected from the alleyway between Buildings C-20, C-21, and C-22 (Figure 3).

  There is no MTCA Method A CUL for cis-1,2-DCE. The detected concentration of cis-1,2-DCE (0.32 mg/kg) in the sample from LAI-25 at a depth of 15 ft bgs exceeded the Method B screening level (0.078 mg/kg – based on the protection of groundwater as drinking water).

The 2017 Phase II soil investigation identified petroleum hydrocarbons and TCE at concentrations greater than the MTCA Method A and B screening levels.

3.2.3 2017 Phase II Soil Gas Investigation Results

Soil gas samples were collected from 25 locations within the TECT lease area and submitted for analysis to Eurofins Air Toxics, of Folsom, California for analysis for VOCs. These sampling locations are identified as LAI-01 through LAI-28 on Figure 3.

VOCs were detected at 21 of 25 sampling locations at concentrations greater than the MTCA Method B shallow soil gas screening level (see Section 5.3) and at 15 of 25 sampling locations at concentrations greater than the Method C shallow soil gas screening level. Exceedances of MTCA Method B and C shallow soil gas screening levels are summarized below.

- 1,1-DCA was detected at a concentration of 64 micrograms per cubic meter (µg/m^3) at LAI-23; this exceeds the Method B shallow soil gas screening level of 52 µg/m^3, but is less than the Method C shallow soil gas screening level of 521 µg/m^3.
- 1-3-butadiene was detected in nine samples at concentrations ranging from 3.2 µg/m^3 to 150 µg/m^3. The detected concentrations exceed the Method B shallow soil gas screening level of 2.8 µg/m^3. The detected concentrations in six of the samples also exceeded the Method C shallow soil gas screening level of 28 µg/m^3.
- Benzene was detected in eight samples at concentrations ranging from 12 µg/m^3 to 73 µg/m^3. The detected concentrations exceed the Method B shallow soil gas screening level of 11 µg/m^3 but are less than the Method C shallow soil gas screening level of 107 µg/m^3.
- Chloroform was detected in eight samples at concentrations ranging from 7.2 µg/m^3 to 64 µg/m^3, which exceed the Method B shallow soil gas screening level of 3.6 µg/m^3. The detected concentration in one sample also exceeds the Method C shallow soil gas screening level of 36 µg/m^3.
- TCE was detected in 13 samples at concentrations ranging from 29 µg/m^3 to 74,000 µg/m^3, which exceed the Method B shallow soil gas screening level of 12 µg/m^3. At eight sampling locations, the detected concentrations also exceed the Method C shallow soil gas screening level of 67 µg/m^3.
- Vinyl chloride was detected in six samples at concentrations ranging from 31 µg/m^3 to 5,400 µg/m^3, which exceed the Method B shallow soil gas screening level of 9.3 µg/m^3. The detected concentrations at three sampling locations also exceeded the Method C shallow soil gas screening level of 93 µg/m^3.

VOCs were detected at concentrations greater than the Method B and Method C shallow soil gas screening levels at locations throughout the TECT lease area. The highest concentrations of VOCs were detected in samples collected from beneath and outside the southwest corner of Building C-22 and from beneath the north end of Building C-23 (the Annex).

3.2.4 2017 Phase II Indoor Air Investigation Results (Building C-23)

In response to the soil gas sampling results, the County requested that LAI conduct indoor air sampling at the currently occupied buildings within the TECT lease area to determine if chemicals detected in soil gas are also present in indoor air at concentrations indicating a potential health concern for buildings occupants. At the time of the indoor air sampling and in preparation for termination of its lease, TECT had moved all business operations to Building C-23 and Buildings C-20, C-21, and C-22 were not occupied (used by TECT for storage). Therefore, indoor air sampling was conducted only at Building C-23. The indoor air evaluation focused on the northern portion of Building C-23 (known as the Annex) where elevated VOC concentrations in soil gas were detected below the building slab and adjacent to the building.

In preparation for indoor air sampling, a survey of Building C-23 was conducted on November 13, 2017. The building survey consisted of observing relevant features of the building construction (e.g., foundation type and condition); documenting the building heating, cooling, and ventilation system; documenting building operations; and conducting a chemical inventory.

Based on the results of the soil gas investigation and the building survey, six indoor air sampling locations were identified (identified on Figures 3 and 7d as IA01-C23 through IA06-C23). Both short-term (8-hour) and long-term (21-day; indicated by "R" in sample name on Figure 3 and 7d) indoor air samples were collected from each of the six locations. An ambient air sample was also collected from an upwind location outside the building during the 8-hour sampling period.

During the 8-hour sampling event, TCE was detected in two indoor air samples at estimated time-weighted average concentrations of 0.2 and 0.21 $\mu g/m^3$. TCE was also detected in the 8-hour ambient air sample at an estimated concentration of 0.31 $\mu g/m^3$, which is higher than the concentrations detected in indoor air. Vinyl chloride was not detected in any of the 8-hour samples at concentrations greater than the laboratory reporting limit.

During the 21-day sampling event, TCE was detected in each of the six indoor air samples at time-weighted average concentrations ranging from 0.40 to 1.7 $\mu g/m^3$. None of the detected concentrations exceeded either the chronic or acute screening criteria for industrial properties. The data indicate that vapor intrusion is likely occurring, but that contaminant concentrations in indoor air are, on average, less than the screening criteria. The data also indicate that contaminant concentrations in indoor air may at least partially be impacted by contaminants present in ambient air.

The historical investigation data described above were considered in developing the planned RI investigation locations discussed in Section 6.0 and shown on Figures 6d and 7e.

3.3 Former Building C-29

This section includes relevant findings from previous Phase I ESAs and subsequent investigations at Building C-29. Building C-29 was included in the Phase I ESA completed by LAI on behalf of the County in 1993, although the assessment report does not identify the building by number. The building was occupied by All Fab, Inc. at the time of the assessment. Relevant finding from the Phase I ESA for Building C-29 are as follows (LAI 1993):

- Building C-29 is described as two interconnected buildings that were used for large-scale chemical storage. One of the buildings contained approximately 100 storage drums, most of which contained petroleum lubricants. Petroleum spillage was observed in this room; however, the underlying concrete did not appear to be degraded. The other building stored several hundred smaller containers (2 gallons or less) of paints and similar compounds. A sump and floor drains were observed in the building; however, there was no information provided regarding the specific location or outlet of the sump or floor drains (LAI 1993). The floor was observed to be very clean, as though recently steam-cleaned. The 1993 report indicated that an inventory of materials contained in the buildings had recently been completed and that disposal of the materials was pending.

Former Building C-29 was demolished by the County in 1996. During demolition, the County discovered a greenish-yellow tinted water at the foundation of the building. The County stopped the demolition work and retained LAI to collect a sample of the water for laboratory analysis (sampling location identified as AF-1 on Figure 3). LAI reported that analysis of the water sample detected chromium, TCE, and vinyl chloride at levels exceeding MTCA drinking water standards (Snohomish County Airport 1996). In July 1996, LAI reported the results of further investigation completed in the vicinity of Building C-29. The study was conducted on behalf of Snohomish County Public Works, based on the January 1996 foundation water sample analysis. Eight test pits were excavated (C29-TP1 through C29-TP8), two soil borings drilled (C29-B3 and C29-B4), and two additional soil borings were drilled and completed as monitoring wells (C29-MW1 and C29-MW2). Two existing monitoring wells were also sampled (SCPWD-1 and SCPWD-2). Analysis of soil and groundwater samples indicated that TCE was present at concentrations exceeding MTCA Method A CULs in soil and groundwater; cis-1,2-DCE was detected at concentrations above MTCA Method B CULs; and vinyl chloride was detected above MTCA Method A CULs in groundwater. Total chromium was detected at a concentration above the MTCA Method A CUL in one soil sample collected from within the building footprint (LAI 1996).

In 1999, AGI conducted a preliminary contamination assessment of Buildings C-19 (described in Section 3.1) and C-29, on behalf of the Snohomish County Public Works Department. AGI identified and collected groundwater samples from eight monitoring wells near Building C-29 that were previously installed by LAI (C29-MW1 and C29-MW2), Snohomish County Public Works Department (SCPWD-1), BF Goodrich or Tramco (HMB1), and AGI (MW1 through MW4). Well depths ranged from 17 to 23 ft bgs and depths to groundwater during sampling ranged from 1.26 ft bgs to 8.26 ft bgs. TCE was detected at a maximum concentration of 18,000 µg/L and the detected concentrations exceeded the MTCA Method A CUL in water samples from seven of the eight wells near Building C-29. Detected concentrations of vinyl chloride exceeded CULs in water from six of the eight wells, and concentrations of 1,2-DCA exceeded CULs in three of the eight wells (AGI 1999).

During CDM's 2000 deep aquifer investigation, discussed in Section 3.5, monitoring well DW2 was installed north of Building C-29. Aquifer groundwater was encountered at approximately 134 ft bgs and chlorinated VOCs (1,1,2-TCA; 1,2-DCA, and 1,2-dichloropropane [DCP]) were detected in groundwater samples collected from DW2 at concentrations exceeding applicable MTCA Method A and B CULs. No cleanup has been conducted at Building C-29 (Rardin 2017).

3.4 Former East Fuel Farm

Environmental investigations at the former East Fuel Farm have been completed on behalf of the US Army Corps of Engineers (USACE) and on behalf of Snohomish County. In 1992, a 12,000 gallon aviation fuel UST (Tank 95B) was removed by a contractor on behalf of the USACE. At the time of the UST removal, approximately 100 cubic yards of petroleum-contaminated soil was removed from the UST excavation and stockpiled on Airport property prior to disposal. Based on the analytical results from confirmation soil samples collected from the remaining soil, contamination exceeding MTCA Method A CULs was not present and no further action was recommended. Records indicate that the USACE planned to remove a second UST (Tank 95; size and contents unknown) at the time of the removal of the 12,000-gallon UST; however, the second UST could not be located (AGI 1993).

In 1994, AGI completed a subsurface investigation at the East Fuel Farm on behalf of Snohomish County. The investigation included a geophysical survey and sampling and analysis of soil and perched groundwater. The results of the 1994 investigation are summarized as follows (AGI 1994):

- The geophysical survey identified two USTs and a potential third UST in the fuel farm. Details regarding the USTs are provided in Section 2.4.5 and the locations of the current and former USTs are shown on the Figure 7 series.
- Ten soil borings (B14-B23) were advanced in the fuel farm using a hollow-stem auger. The soil borings encountered approximately 2 to 17 ft of fill underlain by till. Perched groundwater was encountered at four locations (B16, B20, B21, and B23) at depths ranging from 15 to 22 ft bgs; these borings were completed as monitoring wells MW1 through MW-4. The sampling locations are shown on Figure 3.
- Analytical results for soil samples identified gasoline-range and JP8 jet fuel-range petroleum hydrocarbons in soil at concentrations exceeding the applicable MTCA Method A CULs. AGI concluded that the soil contamination was limited to the UST backfill.
- Analytical results for groundwater identified petroleum hydrocarbons; Jet-A fuel; benzene, toluene, ethylbenzene, and xylenes (BTEX); and chromium in groundwater at concentrations greater than the applicable MTCA Method A CULs.

In 2000, CDM completed an assessment of contamination at the East Fuel Farm on behalf of the County. No additional sampling was completed as part of the assessment. The assessment report indicates that the 25,000-gallon UST was drained and rinsed in 1996 and had not been used since that time (CDM 2000b).

An additional assessment was completed by CDM in 2002 on behalf of the County. The assessment included sampling and analysis of groundwater samples from existing shallow monitoring wells MW-1 through MW-4, and from aquifer well DW-2. Analytical results are summarized as follows (CDM 2002):

- Detected concentrations of petroleum hydrocarbons in samples from MW-1 through MW-3 were below MTCA Method A CULs.
- Gasoline- and diesel-range petroleum hydrocarbons were detected at concentrations greater than the Method A CULs in the sample from MW-4.
- Benzene was detected in each of the wells at concentrations greater than the MTCA Method A CUL.
- Petroleum hydrocarbons and BTEX were not detected in the aquifer sample collected from DW-2.

The 2002 CDM report also indicates that samples were collected from the monitoring wells at the East Fuel Farm in 1996 and 1999 as part of separate investigations conducted at the All Fab facility Building C-29. Chlorinated VOCs were detected in each of the wells indicating potential commingled groundwater plumes in this area. Additional details regarding the C-29 investigations are provided in Section 3.3.

The historical investigation data described above were considered in developing the planned RI investigation locations discussed in Section 6.0 and shown on Figure 8d.

3.5 Deep Aquifer

In 2000, CDM (formerly AGI) completed an investigation of potential contamination of the deep aquifer underlying the Site on behalf of Snohomish County. CDM installed wells to the southwest of Building C-19 (DW1), north of the former Building C-29 (DW2), and northeast of Building C-19 (DW3). The locations of the wells are shown on Figure 3. The wells ranged in depth from 117 ft bgs to 151 ft bgs. Soil samples were collected at 10-ft intervals during drilling, field-screened for VOCs and laboratory-analyzed for halogenated VOCs. Groundwater samples were collected from DW1 and DW2 in December 1999 and March 2000 and from DW3 in May 2000. Additional groundwater samples were collected from monitoring well DW1 in 2003 and analyzed for halogenated VOCs.

- At monitoring well DW1, aquifer groundwater was encountered at approximately 133 ft bgs. Analysis of groundwater samples from DW1 indicated that TCE had migrated vertically to the aquifer. TCE and cis-1,2-DCE were detected in aquifer groundwater samples from DW1, but only TCE (maximum detected concentration of 62 µg/L in 2000) exceeded applicable MTCA Method A or B CULs. TCE was detected at a concentration above the reporting limit in one soil sample (57.8 ft bgs), but below the MTCA Method A and B CULs (CDM 2000a). CDM's analysis of groundwater samples from DW1 in 2003 indicated TCE concentrations in the aquifer were increasing. TCE was detected at a concentration of 81 µg/L in a sample collected from DW1 in 2003.
- At monitoring well DW2, aquifer groundwater was encountered at approximately 134 ft bgs. TCE was not detected in groundwater samples collected from DW2; however, concentrations of 1,1,2-TCA, 1,2 DCA, and 1,2-DCP were detected above MTCA Method A and B CULs in the groundwater samples. TCE was detected at a concentration above the MTCA Method B CUL for protection of groundwater in one soil sample. Trans-1,2-DCE and cis-1,2-DCE were

detected at concentrations below applicable CULs in soil samples collected from DW2 (CDM 2000a).

- At monitoring well DW3, aquifer groundwater was encountered at approximately 132 ft bgs. VOCs were not detected in soil or groundwater samples from DW3.

CDM concluded that the data did not suggest the presence of area-wide contamination of the aquifer; however, following an analysis of the degradation pathways expected of the known products used by All Fab and the historical disposal practices, anomalies were noted that could indicate a contaminant source other than the known releases near Buildings C-19 and C-29 (CDM 2000a). However, as noted in Section 3.1.2, further investigation completed in 2005 did not identify a TCE source other than Building C-19 (CDM 2005).

The historical investigation data described above were considered in developing the planned RI investigation locations discussed in Section 6.0 and shown on Figure 9c.

4.0 PRELIMINARY CONCEPTUAL SITE MODEL

This section describes the geology and hydrogeology of the Site based on information developed during previous investigations. Further information regarding geology and hydrogeology at the Site will be obtained during the RI and described in the RI report, and will include hydrogeologic cross sections developed from the borings advanced during the RI and existing information, and groundwater elevation contour maps developed from the monitoring of the wells installed during the RI.

This section presents a preliminary conceptual site model that identifies potential contaminants of concern (PCOCs) at the Site, areas at the Site that have the greatest potential to be sources of contaminants, and the potential contaminant migration pathways and receptors. A schematic of the conceptual site model will be developed and included in the RI report.

4.1 Geology

The Puget Sound region is underlain by Quaternary sediments deposited by numerous glacial episodes, the most recent of which is termed the Vashon Stade of the Fraser Glaciation. Deposition occurred during a number of glacial advances and retreats. The last cycle of glacial advance and retreat resulted in the present-day topographic expression of the area, many of the near-surface deposits, and existing subsurface conditions. The glacial sediments, in general order from most shallow to deepest, are made up of interlayered and sequential deposits of glaciomarine drift, glacial recessional outwash, glacial till, and glacial advance outwash. The glacial till and underlying units have been overconsolidated due to the weight of the overriding ice sheets. These glacial sediments are overlain with more recent, non-glacial deposits locally consisting of beach sands and gravels; alluvial silt, sands, and gravels; and/or lake clays, silts, and peat that were deposited following the glacial retreats (Booth et al. 2004).

Previous investigations conducted at the Site and adjacent properties encountered fill material to depths of 0 to 20 ft bgs, underlain by Vashon Till, which is composed of a very dense, heterogeneous mixture of gravel, sand, silt, and clay, with localized sand lenses. The till at the Site is up to 80 ft thick and is underlain by Vashon Advance Outwash, which is a gray, fine to medium sand with gravel that extends to approximately 100 ft bgs and then transitions to a fine to medium sand to approximately 145 ft bgs, where the sand becomes gravelly (CDM 2000a).

4.2 Hydrogeology

Based on the results of previous investigations at the Site, perched groundwater occurs at depths as shallow as 1.5 to 15.5 ft bgs. The direction of perched groundwater flow was observed to vary seasonally, toward the east or southeast (CDM 2005). Recent investigations (LAI 2018) have shown that the perched groundwater encountered historically is not present during the dry months of the year at most areas of the Site, thus the perched water should be considered seasonal.

A groundwater aquifer was encountered in the Vashon Advance Outwash at approximately 130 to 135 ft bgs. Groundwater flow in the aquifer was observed to be to the north to northeast (CDM 2000a, 2005).

4.3 Potential Contaminants of Concern and Sources

Site PCOCs are designated based on the contaminants associated with the potential sources identified in Section 3.0 that have been detected during previous investigations or remedial actions at the Site, or that are suspected to be present based on available information. The PCOCs consist primarily of VOCs, metals (MTCA [arsenic, cadmium, chromium, lead, and mercury]) and hexavalent chromium) and petroleum hydrocarbons. The specific PCOCs for each area being investigated under the AO are as follows:

- PCOCs at Building C-19 include:
    - VOCs
    - Metals (MTCA metals, in particular chromium (III and VI)]
    - Petroleum hydrocarbons (diesel-range organics [DRO]/oil-range organics [ORO])
- PCOCs at former Building C-29 include:
    - VOCs
    - Metals [MTCA metals, in particular chromium (III and VI) and lead]
    - Petroleum hydrocarbons [gasoline-range organics (GRO), DRO, ORO]
- PCOCs at the East Fuel Farm include:
    - VOCs
    - Metals [MTCA metals, in particular chromium (III and VI) and lead]
    - Petroleum hydrocarbons (GRO, DRO, ORO)
- PCOCs at Building C-23 include:
    - VOCs
    - Metals (MTCA metals, in particular lead and chromium)
    - Petroleum hydrocarbons (DRO, ORO)
- PCOCs at the Building C-20, C-21, C-22 complex include:
    - VOCs
    - Metals (MTCA metals, in particular lead and chromium)
    - Petroleum hydrocarbons (DRO, ORO)
- PCOCs for the deep aquifer include:
    - VOCs.

Although extensive investigation work has already been conducted at the Site, a review of historical data indicates that analysis for polychlorinated biphenyls (PCBs) has not been conducted at the Site. The Site history indicates that additional sample analysis for PCBs in soil is warranted in locations where releases of hydraulic oil has occurred, as historically PCBs were a common component of hydraulic oils. PCBs are therefore also considered a PCOC and will be included for analysis as appropriate in areas of the Site where hydraulic oil was used or stored.

4.4 Contaminant Media of Potential Concern, Contaminant Migration, Receptors, and Exposure Pathways

As described in Section 4.1 and 4.2, the Site geology consists of a relatively thin layer of fill material underlain by glacial till with advance outwash underlying the glacial till. Seasonal perched groundwater is present above the glacial till in some areas of the Site. The regional aquifer is located at approximately 130 ft bgs in the advance outwash.

Much of the Site surface is paved, with some unpaved areas to the northwest, north, and east. No surface water features are located near the Site and stormwater runoff is collected in the onsite stormwater conveyance system or runs off and infiltrates in unpaved areas.

The Site and the surrounding area are highly urbanized and are part of an airport facility where use by wildlife is actively discouraged due to aviation safety concerns. Therefore, wildlife are not identified as potential receptors and a terrestrial ecological evaluation is not proposed as part of the RI. The Site is zoned for industrial land use and the primary use of the Site is industrial; the primary receptors are adult workers. Adult workers include both people working at the Site long-term and temporary construction workers.

4.4.1 Exposure Pathways

Previous investigations indicate that soil, groundwater, and soil gas contamination is present in the upper glacial till and fill layers and groundwater contamination is present in the regional aquifer. Potential exposure and migration pathways are summarized in the following subsections by medium.

4.4.1.1 Soil

Potential exposure and migration pathways in shallow soil include:

- Direct contact including incidental ingestion or dermal contact by Site workers with hazardous substances that are present in subsurface soil or inhalation of hazardous substances that are present in soil that have migrated as windblown or fugitive dust during construction activities
- Leaching of contaminants from soil to groundwater
- Leaching of contaminants from soil to stormwater and ultimately surface water.

4.4.1.2 Groundwater

Potential exposure and migration pathways for shallow groundwater include:

- Incidental ingestion and dermal contact by construction workers of hazardous substances that are present in groundwater
- Migration of contaminated shallow groundwater to the regional aquifer
- Partitioning of contaminants from groundwater to shallow soil gas.

Potential exposure pathways for regional groundwater include:

- Ingestion as drinking water of hazardous substances that are present in groundwater.

4.4.1.3 Soil Gas/Indoor Air

Potential exposure pathways for soil gas include:

- Migration of hazardous substances in soil gas to indoor air
- Inhalation of hazardous substances in indoor air by workers.

5.0 SCREENING LEVELS

Screening levels are tools used to help remediation professionals and regulators evaluate RI data and are typically established as concentrations for each compound and medium of potential concern at a site. Screening levels are typically established at the most conservative regulatory concentrations in order to ensure that appropriate analytical methods are employed. Screening levels are not cleanup levels; cleanup levels are typically developed during the Feasibility Study (FS) phase of the project and are often different than the screening levels for a particular compound or medium. Cleanup standards, also typically developed during the FS phase, include cleanup levels; the point of compliance, the location where cleanup levels must be met; and other regulatory requirements that apply to a site.

Soil, groundwater, and air screening levels were developed based on the media and exposure pathways identified in Sections 4.3 and 4.4 and MTCA requirements, as described below.

5.1 Soil Screening Levels

Soil screening levels were developed for unrestricted land use in accordance with WAC 173-340-740 using MTCA Method B. Although it appears the Site will meet the MTCA definition for an industrial property, a determination has not yet been made that industrial land use represents the reasonable maximum exposure. Basing screening levels on unrestricted land use provides a conservative evaluation of constituents for initial screening of data and addresses potential exposure pathways including potential exposure of future receptors if the Site use changes and protection of groundwater that could potentially be used as drinking water in the future. Under MTCA Method B, soil cleanup levels must be at least as stringent as all of the following[1]:

- Concentrations established under applicable state and federal laws
- Concentrations protective of human health:
    - Concentrations that, due to direct contact with contaminated soil, are estimated to result in no acute or chronic non-carcinogenic or carcinogenic toxic effects on human health
    - Concentrations that will not cause contamination of groundwater at levels which exceed groundwater cleanup levels.

These criteria were considered during development of soil screening levels. Soil screening levels were developed for all constituents detected in previous soil or groundwater investigations and in some cases for additional compounds that are part of the same chemical class or that have not been analyzed at the Site but are planned for analysis during the RI. Soil screening levels are provided in Table 12.

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[1] Protection of ecological receptors was not considered because the Site is believed to meet the exemption criteria for a terrestrial ecological evaluation.

The only constituent for which concentrations have been established under other applicable state and federal laws besides MTCA are PCBs. PCB cleanup levels are established under the Toxic Substances Control Act (TSCA) for high occupancy uses at 1.0 mg/kg. Therefore, for PCBs, the screening levels were set at the lowest of the TSCA cleanup level and the concentration protective of direct human contact. Except for TPH, mercury, and lead, standard MTCA Method B soil screening levels protective of direct human contact were determined in accordance with WAC 173-340-740(3) using Ecology's Cleanup Levels and Risk Calculations (CLARC) database (Ecology 2015). MTCA Method A soil cleanup levels for unrestricted land uses were used for TPH, mercury, and lead.

Soil screening levels protective of groundwater were determined using the fixed-parameter three-phase partitioning model in accordance with WAC 173-340-747(4) as identified in Ecology's CLARC database[2] (Ecology 2015). Groundwater screening levels used in calculating contaminant concentrations in soil protective of groundwater were developed as described in Section 5.2. The three-phase model provides a conservative estimate of the concentration of a contaminant in soil that is protective of groundwater.

The CLARC database does not provide a screening level for total chromium. Therefore, the screening level for total chromium was set at natural background levels published by Ecology (1994).

5.2 Groundwater Screening Levels

Groundwater screening levels were developed for detected constituents in previous soil or groundwater investigations using the standard MTCA Method B [WAC 173-340-720(4)]. Groundwater at the Site is not used as drinking water; however, to provide a conservative evaluation of constituents and to address potential future use of groundwater as drinking water, screening levels were based on drinking water as the highest potential beneficial use for groundwater. Under MTCA Method B, groundwater cleanup levels must be at least as stringent as all of the following[3]:

- Concentrations established under applicable state and federal laws
- Concentrations protective of human health determined using MTCA Equations 720-1 or 720-2, if sufficiently protective health-based criteria have not been established under applicable state and federal laws.

Screening levels were established based on these cleanup level requirements. Although MTCA allows for a maximum carcinogenic risk of $1x10^{-5}$ for constituents for which maximum contaminant levels (MCLs) have been established under applicable state or federal laws, screening levels were based on a maximum carcinogenic risk of $1x10^{-6}$. Screening levels were set at the lowest of the federal and state MCLs, if applicable, and the MTCA Method B formula value (calculated using MTCA Equation 720-1 for non-carcinogens and Equation 720-2 for carcinogens). Groundwater concentrations protective of the

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[2] Soil-to-groundwater cleanup levels for saturated soil were calculated using Henry's constants at 13°C.

[3] Contaminant concentrations must be protective of surface water beneficial uses if impacted groundwater will reach surface water; however, no surface water features are located near the Site.

vapor intrusion pathway were also considered for compounds that are proposed for analysis in soil gas (see Section 5.3). If no federal or state criteria were available, the lowest of the MTCA Method B formula values were used as the screening criterion. Groundwater screening levels were developed for all constituents detected in previous soil or groundwater investigations and in some cases for additional compounds that are part of the same chemical class or that have not been analyzed at the Site but are planned for analysis during the RI. Groundwater screening levels are provided in Table 13.

5.3 Air/Vapor Intrusion Screening Levels

Screening levels protective of the vapor intrusion pathway were established for indoor air, soil gas, and groundwater based on the standard MTCA Method B [173-340-750(3)]. Indoor air screening levels were calculated using equations 750-1 for non-carcinogens and 750-2 for carcinogens. Soil gas and groundwater screening levels protective of indoor air were calculated using the Method B indoor air values and appropriate attenuation factors and Henry's Law Constants as established in Ecology's CLARC database. Soil gas screening levels are provided in Table 14.

Because air samples are susceptible to interference from background sources, the list of analytes was narrowed based on the results from previous soil gas investigations. Previous data were screened against soil gas screening levels provided in the CLARC database. Of the six detected compounds that exceeded the screening levels in the CLARC database, two compounds (chloroform and 1,3-butadiene) were excluded because they are considered background contaminants, and historical information does not indicate that the compounds were used or released at the Site. In addition, two compounds (tetrachloroethene and 1,1,1-TCA) were added to the air analyte list because historical information indicates that they may have been used at the Site and analysis of these compounds in soil gas will help determine if a release occurred.

6.0 REMEDIAL INVESTIGATION

Further investigation of soil, groundwater, and soil gas is needed to evaluate the nature and extent of contamination in each of the investigation areas identified based on previous investigations at the Site. Data from the RI will be used to determine if cleanup is warranted, and, if warranted, to develop and evaluate cleanup action alternatives and select a final cleanup action.

The RI field investigation will be completed in phases to collect the data required to assess the nature and extent of any releases of PCOCs. Additional details are provided in the following sections; however, the general sequence of work is anticipated to be as follows:

- Collection and analysis of groundwater samples and utility locating. This phase of the investigation will include:
    - Evaluation of the condition of existing monitoring wells and redevelopment and repair of existing monitoring wells, as needed
    - Collection and laboratory analysis of groundwater samples from existing shallow and deep monitoring wells
    - Completion of utility locating to confirm locations of mapped utilities and identify unknown or unmapped subsurface utilities and other features such as USTs.
- Collection and laboratory analysis of soil, groundwater grab, and soil gas samples from new soil borings in each of the investigation areas and installation and sampling of three new deep monitoring wells. This phase of investigation will include:
    - Collection of soil samples from new soil borings in each investigation area
    - Collection of groundwater samples from selected soil borings, if groundwater is encountered
    - Collection of soil gas samples from selected soil borings and from additional soil borings where collection of groundwater grab samples is planned, but groundwater is not encountered
    - Installation of three deep aquifer monitoring wells.
- Installation and sampling of shallow monitoring wells in each investigation area based on the analytical results for groundwater samples collected from existing monitoring wells and the analytical results for the groundwater grab samples.
- Installation of additional deep monitoring wells and collection and laboratory analysis of supplemental soil, groundwater, and soil gas samples to fill data gaps remaining after previous phases of investigation.
- Completion of a second round of groundwater monitoring from existing and newly installed monitoring wells to evaluate seasonal variations in groundwater conditions.

This section presents the proposed scope of the RI. Proposed RI activities are also summarized in Table 16. More detail for each proposed RI activity is provided in the SAP (Appendix A). The scope of the later stages of investigation will be adjusted, if warranted, based on the results from initial stages of investigation.

During the RI activities, if any archaeological resources are discovered, work will be stopped immediately and Ecology, the Washington State Department of Archaeology and Historic Preservation (DAHP), Snohomish County, and the appropriate Tribes' cultural resources departments will be notified by the close of business on the day of discovery. A licensed archaeologist will inspect the site and document the discovery, provide a professionally documented site form, and report to the above-listed parties. In the event of an inadvertent discovery of human remains, work will be immediately halted in the discovery area, the remains will be covered and secured against further disturbance, and the Snohomish County Police Department and Snohomish County Medical Examiner will be immediately contacted, along with the DAHP physical anthropologist and authorized Tribal representatives. A treatment plan by a licensed archaeologist would then be developed in consultation with the above-listed parties consistent with Chapters 27.44 and 27.53 RCW and implemented in accordance with Chapter25-48 WAC.

6.1 Existing Monitoring Wells

Shallow and deep monitoring wells were installed at the Site during previous investigations, as described in Section 3.0. Based on conditions observed at the time of a July 2018 site walk, redevelopment and repair of some of these wells will be needed prior to sampling. Additional information regarding the construction and condition of the existing shallow monitoring wells, along with recommended measures to recondition (e.g., redevelop) or repair the wells, is provided in Table 15.

6.1.1 Shallow Monitoring Wells

The following shallow monitoring wells remain at the Site:

- Building C-19: Shallow monitoring wells SCPWD-2, SCPWD-3, and SCPWD-4 are present near the south end of Building C-19.
- Former Building C-29/Former East Fuel Farm: Shallow monitoring wells MW-1, MW-2, MW-3, MW-4, and HMB1 are present in the vicinity of the former East Fuel Farm. Shallow monitoring wells SCPWD-1, C29-MW1, and C29-MW2 are present in the vicinity of former Building C-29.

Groundwater samples will be collected from each of the shallow monitoring wells and analyzed in accordance with the sampling schedule presented in Table 16. Groundwater sample analyses will include VOCs, DRO/ORO, GRO, dissolved MTCA metals, 1,4-dioxane, and natural attenuation parameters.

6.1.2 Deep Monitoring Wells

Three deep monitoring wells (DW1, DW2, and DW3) are present at the Site from previous investigations as described in Section 3.1.4. Groundwater samples will be collected from each of the deep monitoring wells and analyzed in accordance with the sampling schedule presented in Table 16. Groundwater sample analyses will include VOCs, 1,4-dioxane, and natural attenuation parameters.

6.2 Building C-19

This section identifies the data gaps, RI objectives and planned RI activities for the Building C-19 investigation area.

6.2.1 Data Gaps and RI Objectives

The following data gaps have been identified for Building C-19:

- VOCs have been detected in soil and groundwater at the south end of the building, in the area of the former vapor degreaser at concentrations greater than applicable screening levels. The contamination extends to the southwest of the building and is potentially migrating along storm and sanitary sewer lines. The extent of soil and shallow groundwater has not been fully delineated.
- Investigation and cleanup of TPH-D and TPH-O in soil beneath the floor of the manufacturing areas of the buildings was completed between 2006 and 2012; Ecology issued an NFA determination for the cleanup in 2013. Available information indicates that chlorinated cutting fluids were historically used at the Site and that metals are a potential COC for this investigation area. Sampling for VOCs or metals was not conducted as part of the previous investigation and cleanup.
- Heavy petroleum staining was previously observed in a former drum storage area on the east side of the building. No investigation has been completed in this area.
- The potential for vapor intrusion into Building C-19 was not evaluated as part of previous investigations.

The objectives of the RI are to:

- Delineate the extent of chlorinated VOCs in soil and shallow groundwater
- Identify potential migration pathways in the vicinity of the former vapor degreaser
- Evaluate the potential for metals and petroleum hydrocarbons to be present in soil and groundwater
- Evaluate potential vapor intrusion into Building C-19.

Previous investigations indicate that TCE has migrated vertically to the aquifer, and that concentrations in the aquifer were increasing. Planned RI activities associated with the deep aquifer are outlined in Section 6.5.

6.2.2 Soil Investigation

The soil investigation will consist of collecting soil samples from soil borings advanced at the locations shown on Figure 5d, which will be submitted for laboratory analysis as described in Table 16. The explorations will consist of the following:

- Four soil borings (RISB-4, RISB-5, RISB-54, and RISB-55) will be advanced inside Building C-19 to the north and northeast of the former vapor degreaser. Four additional soil borings will be advanced outside of the building to the east (RISB-3), west (RISB-11), northeast (RISB-6), and northwest (RISB-7) of the former degreaser and in the vicinity of historical industrial operations, including the former drum storage area on the east side of the building.
- Five soil borings (RISB-1, -2, -8, -9, and -10) will be advanced in a halo approximately 150 ft beyond the south end of Building C-19 to delineate the extent of previously identified contamination.
- As described on Table 16, soil sample analyses will include VOCs, DRO/ORO, MTCA Metals, and PCBs. Two soil samples will also be selected from the above-described soil borings for analysis for total organic carbon (TOC) and grain size.

At each proposed exploration location, soil samples will be collected from borings advanced using either a direct-push drilling rig or a mini-sonic drilling rig. Each boring will be advanced to approximately 5 ft below the fill-till interface unless contamination is observed through field screening at the bottom of the 5-ft penetration into the till, in which case the boring will continue to be advanced until contamination is no longer observed. Soil sampling and analysis procedures are discussed in more detail in the SAP (Appendix A).

6.2.3 Groundwater Investigation

As described above, the groundwater investigation will be completed in phases. Samples will be collected from existing monitoring wells prior to drilling new soil borings. In addition to the three existing shallow monitoring wells at Building C-19, groundwater grab samples will be collected from up to 12 of the soil borings described in Section 6.2.2. The anticipated locations of the groundwater grab samples are shown on Figure 5d. The final locations for the groundwater grab samples may be adjusted based on the results from sampling of existing monitoring wells. The final locations of groundwater grab samples will also be determined by the locations at which groundwater is encountered during drilling.

The final phase of groundwater investigation at Building C-19 will consist of the installation of up to three shallow monitoring wells inside Building C-19 and up to five additional shallow monitoring wells outside the building. The locations of the shallow monitoring wells will be determined based on the results from the initial phases of groundwater investigation described above. The proposed well locations will be provided to the County for review prior to installation.

The sampling and analysis plan for groundwater is outlined in Table 16. Groundwater grab samples from borings will be analyzed with an expedited turnaround time for decision-making regarding monitoring well installation. Groundwater sample analyses will include VOCs, DRO/ORO, dissolved MTCA metals, 1,4-dioxane, and natural attenuation parameters in accordance with Table 16. Groundwater samples from newly installed monitoring wells will be analyzed for VOCs. Additional analysis may be conducted based on the results of analysis of groundwater samples from existing monitoring wells and groundwater grab samples.

6.2.4 Soil Gas and Indoor Air Investigation

The sampling and analysis plan for soil gas is outlined in Table 16. As indicated, soil gas samples will be collected from four locations within the south end of Building C-19 (RISB-4, and -5). Additional soil gas samples may be collected from areas where groundwater grab samples are planned (see Section 6.2.3), but where shallow perched water is not encountered. Soil gas samples will be analyzed for TCE and related compounds.

Indoor air samples will be collected from Building C-19 if VOCs are detected in soil gas samples or shallow groundwater samples collected beneath or adjacent to the building at concentrations greater than the MTCA Method B screening levels based on vapor intrusion. Up to two indoor air samples and one ambient air sample will be collected over an 8-hour time period. The samples will be analyzed for only those VOCs that are detected in soil gas or shallow groundwater at concentrations greater than the screening levels for vapor intrusion.

6.3 Building C-20, C-21, C-22 Complex

This section identifies the data gaps, RI objectives and planned RI activities for the Building C-20, C-21, C-22 complex investigation area.

6.3.1 Data Gaps and RI Objectives

The following data gaps have been identified for the Building C-20, C-21, C-22 complex:

- VOCs have been detected in soil and soil gas at the southwest corner of Building C-22, in the area of a former machine pit and sump, at concentrations greater than applicable screening levels. The extent of contamination has not been delineated.
- Groundwater conditions in this area of the Site have not been evaluated.
- The potential for vapor intrusion into the buildings was not evaluated as part of previous investigations.
- Contamination has been identified along the trench drain located within Building C-22 and running east to west along the north side of Building C-20. The extent of contamination has not been delineated and the discharge point of the trench drain is not known.
- Two USTs were reportedly removed from the north side of Building C-22; however, documentation of the removal is not available and limited sampling has been conducted in this area.

The objectives of the RI are to:

- Delineate the extent of chlorinated VOCs and petroleum hydrocarbons in soil along the trench drain

- Determine the discharge point of the trench drain
- Identify the source of VOCs in soil and soil gas at Building C-22 and evaluate potential migration pathways
- Evaluate shallow groundwater in areas of identified soil and soil gas contamination
- Confirm that USTs were removed from the north side of Building C-22 (see Section 6.8.2) and evaluate soil and groundwater conditions in the area of the former tanks.

6.3.2 Soil Investigation

The soil investigation will consist of collecting soil samples from soil borings advanced at the locations shown on Figure 6d, which will be submitted for laboratory analysis as described in Table 16. The explorations will consist of the following:

- Six soil borings (RISB-13 and RISB-24 through RISB-28) will be advanced in the vicinity of the trench drain located along the north side of Building C-20.
- Two soil borings (RISB-49 and RISB-50) will be advanced in the area of the former machine pit and sump in the southwestern corner of Building C-22. Four additional soil borings (RISB-18 through RISB-21) will be advanced outside the southwest corner of the building to delineate the extent of previously identified contamination.
- Three soil borings (RISB-12, RISB-16, and RISB-17) will be advanced near the northwest corner of Building C-22, where two USTs were reportedly removed.
- Two soil borings will be advanced to the north of Building C-21 (RISB-14 and RISB-15) and two soil borings will be advanced to the south of Building C-20 (RISB-23) and C-22 (RISB-22) to evaluate conditions in areas that have not been previously investigated and to further delineate areas of contamination identified during previous investigations.
- Soil samples will be collected for laboratory analysis as described in Table 16. Soil sample analyses will include VOCs, DRO/ORO, MTCA Metals, and PCBs. Two soil samples will also be selected from the above-described soil borings for analysis for TOC and grain size.

At each proposed exploration location, soil samples will be collected from borings advanced using either a direct-push drilling rig or a mini-sonic drilling rig. Each boring will be advanced to approximately 5 ft below the fill-till interface unless contamination is observed at the bottom of the 5-ft penetration into the till, in which case the boring will continue to be advanced until contamination is no longer observed. The soil sampling and analysis procedures are discussed in more detail in the SAP (Appendix A).

6.3.3 Groundwater Investigation

Groundwater conditions in this portion of the Site have not been previously investigated. Groundwater grab samples will be collected from up to 18 of the soil borings described in Section 6.3.2. The anticipated locations of the groundwater grab samples are shown on Figure 6d. Groundwater was not encountered in this portion of the Site during a previous investigation;

therefore, the final locations of groundwater grab samples may be limited by the locations at which groundwater is encountered during drilling.

If groundwater is encountered during drilling of the soil borings described above, the final phase of groundwater investigation at the Building C-20, C-21, C-22 complex will consist of installation of up to three shallow monitoring wells inside the buildings and up to four additional shallow monitoring wells outside the buildings. The locations of the shallow monitoring wells will be determined based on the results from the groundwater grab sampling described above. The proposed well locations will be provided to the County for review prior to installation.

The sampling and analysis plan for groundwater is outlined in Table 16. Groundwater grab samples from borings will be analyzed with an expedited turnaround time for decision-making regarding monitoring well installation. Groundwater sample analyses will include VOCs, DRO/ORO, and dissolved MTCA metals, in accordance with Table 16. Groundwater samples from newly installed monitoring wells will be analyzed for VOCs. Additional analysis may be conducted based on the results of analysis of groundwater samples from existing monitoring wells and groundwater grab samples.

6.3.4 Soil Gas and Indoor Air Investigation

Soil gas sampling is not currently planned at the Building C-20, C-21, C-22 complex. However, soil gas samples will be collected from selected locations where groundwater grab samples are planned (see Section 6.3.3), but where shallow perched water is not encountered. Soil gas samples will be analyzed for TCE and related compounds. The data will be used to evaluate the vapor intrusion pathway for this area of the Site by comparing soil gas data to the MTCA Method B screening levels for evaluating the vapor intrusion pathway.

Indoor air samples will not be collected from Buildings C-20, C-21 or C-22. These buildings are currently unoccupied and will be demolished.

6.4 Building C-23 / C-23 Annex

This section identifies the data gaps, RI objectives and planned RI activities for the Building C-23/C-23 Annex investigation area.

6.4.1 Data Gaps and RI Objectives

The following data gaps have been identified for Building C-23/C-23 Annex:

- VOCs have been detected in soil gas beneath the north end of Building C-23 and the south end of the C-23 Annex at concentrations greater than applicable screening levels. The source and extent of contamination have not been identified and groundwater conditions have not been evaluated.
- Petroleum hydrocarbons were detected in soil beneath the floor in the machining area in the southwestern corner of the building. Limited soil investigation was completed in this area. The

extent of soil contamination has not been delineated and groundwater conditions have not been evaluated.

- Chemical and waste storage areas are located on the east side of the building (oil shed and metal chip processing area) and on the west side of the Annex (liquid waste storage tanks). Limited soil and soil gas sampling has been completed in these areas and groundwater conditions have not been evaluated.
- At least one heating oil UST is located at the southwest corner of the building. Previous reports include conflicting information regarding the number of USTs and a second UST is potentially present. Limited sampling has been conducted in the vicinity of the UST(s) and groundwater conditions have not been evaluated.

The objectives of the RI are to:

- Investigate the source and delineate the extent of chlorinated VOCs beneath the northern portion of Building C-23 and the south end of the C-23 Annex
- Delineate the extent of petroleum hydrocarbons in shallow soil beneath the floor in the machining area
- Determine if one or more USTs are present near the southwest corner of the building (see Section 6.8.2) and evaluate soil and groundwater conditions in the area of the UST(s).
- Evaluate shallow groundwater in areas of identified soil and soil gas contamination.

6.4.2 Soil Investigation

The soil investigation will consist of collecting soil samples from borings advanced at the locations shown on Figure 7e, which will be submitted for laboratory analysis as described in Table 16. The explorations will consist of the following:

- Seven soil borings (RISB-33 through RISB-38 and RISB-53) will be advanced in and around the machining area located in the southwestern portion of the building to delineate the extent of previously identified contamination and to evaluate subsurface conditions in areas that were not included in the previous investigation.
- Two soil borings (RISB-39 and RISB-40) will be advanced in the area of the oil shed and metal chip processing area on the west side of the building.
- Two soil borings (RISB-29 and RISB-51) will be advanced in the northern portion of the building (Annex) where VOCs were previously detected in soil gas at concentrations greater than applicable screening levels.
- Four soil borings (RISB-30 through RISB-32 and RISB-52) will be advanced along the west side of the building, near and downgradient of the liquid waste storage area. Data from these borings will also be used to further delineate areas of contamination identified during previous investigations at former Building C-29, as discussed in Section 6.5.
- As described in Table 16, soil sample analyses will include VOCs, DRO/ORO, MTCA Metals, and PCBs. Two soil samples will also be selected from the above-described soil borings for analysis for TOC and grain size.

At each proposed exploration location, soil samples will be collected from borings advanced using either a direct-push drilling rig or a mini-sonic drilling rig. Each boring will be advanced to approximately 5 ft below the fill-till interface unless contamination is observed at the bottom of the 5-ft penetration into the till, in which case the boring will continue to be advanced until contamination is no longer observed or refusal is met. The soil sampling and analysis procedures are discussed in more detail in the SAP (Appendix A).

6.4.3 Groundwater Investigation

Groundwater conditions in this portion of the Site have not been previously investigated. Groundwater grab samples will be collected from up to 12 of the soil borings described in Section 6.4.2. The anticipated locations of the groundwater grab samples are shown on Figure 7e. Groundwater was not encountered in this portion of the Site during previous investigations; therefore, the final locations of groundwater grab samples may be limited by the locations at which groundwater is encountered during drilling.

If groundwater is encountered during drilling of the soil borings described above, the final phase of groundwater investigation at Building C-23 will consist of installation of up to three shallow monitoring wells inside the buildings and up to five additional shallow monitoring wells outside the building. The locations of the shallow monitoring wells will be determined based on the results from the groundwater grab sampling described above. The proposed well locations will be provided to the County for review prior to installation.

The sampling and analysis plan for groundwater is outlined in Table 16. Groundwater grab samples from borings will be analyzed with an expedited turnaround time for decision-making regarding monitoring well installation. Groundwater sample analyses will include VOCs, DRO/ORO, dissolved MTCA metals, and natural attenuation parameters in accordance with Table 16. Groundwater samples from newly installed monitoring wells will be analyzed for VOCs. Additional analysis may be conducted based on the results of analysis of groundwater samples from existing monitoring wells and groundwater grab samples.

6.4.4 Soil Gas and Indoor Air Investigation

Soil gas sampling is not currently planned at Building C-23. However, soil gas samples will be collected from selected locations where groundwater grab samples are planned (see Section 6.4.3), but where shallow perched water is not encountered. Soil gas samples will be analyzed for TCE and related compounds. The data will be used to evaluate the vapor intrusion pathway for this area of the Site. Indoor air samples will not be collected from Building C-23. Indoor air sampling was completed in November 2017.

6.5 Former Building C-29 / Former East Fuel Farm

This section identifies the data gaps, RI objectives and planned RI activities for the former Building C-29 and former East Fuel Farm investigation area.

6.5.1 Data Gaps and RI Objectives

Previous investigations at former Building C-29 and the former East Fuel Farm have identified chlorinated VOCs, metals, and petroleum hydrocarbons in soil and groundwater. Data from previous investigations suggest that commingled groundwater plumes are present in this area of the Site. The following data gaps have been identified for the former Building C-29 / former East Fuel Farm:

- VOCs have been detected in soil and groundwater within and in the vicinity of the footprint of former Building C-29 at concentrations greater than applicable screening levels. The extent of contamination has not been delineated, but appears to extend to the former East Fuel Farm.
- Petroleum hydrocarbons were detected in soil and groundwater in the former East Fuel Farm. The extent of contamination has not been delineated.
- Chromium is present in soil and groundwater at former Building C-29 and in groundwater at the former East Fuel Farm. Sampling and analysis for hexavalent chromium have not been conducted.
- USTs remain at the former East Fuel Farm. A geophysical survey identified two USTs and a potential third UST in the fuel farm. The tanks were reportedly drained and rinsed. The current condition of the tanks and surrounding soil is not known.

The objectives of the RI are to:

- Investigate the source and delineate the extent of chlorinated VOCs, metals, and petroleum hydrocarbons in soil and groundwater and determine if hexavalent chromium is present.
- Confirm the status of the remaining USTs (see Section 6.8.2).

6.5.2 Soil Investigation

The soil investigation will consist of collecting soil samples from soil borings advanced at the locations shown on Figure 8d, which will be submitted for laboratory analysis as described in Table 16. The explorations will consist of the following:

- One soil boring (RISB-48) within the footprint of former Building C-29
- Seven soil borings (RISB-41 through RISB-47) to the west and north of former Building C-29 and the former East Fuel Farm to delineate the extent of previously identified contamination
- As indicated above, four soil borings (RISB-30 through RISB-32 and RISB-52) will be advanced along the west side of adjacent Building C-23 and data from those borings will be used to evaluate the extent of contamination associated with former Building C-29.
- As described in Table 16, soil samples analyses will include VOCs, DRO/ORO, GRO, MTCA Metals, and PCBs. Two soil samples will also be selected from the above-described soil borings for analysis for TOC and grain size.

At each proposed exploration location, soil samples will be collected from borings advanced using either a direct-push drilling rig or a mini-sonic drilling rig. Each boring will be advanced to approximately 5 ft below the fill-till interface unless contamination is observed at the bottom of the 5-ft penetration into the till, in which case the boring will continue to be advanced until contamination is no longer observed. The soil sampling and analysis procedures are discussed in more detail in the SAP (Appendix A).

6.5.3 Groundwater Investigation

The groundwater investigation in this area will be completed in phases. Samples will be collected from existing monitoring wells prior to drilling new soil borings. In addition to the eight existing monitoring wells, groundwater grab samples will be collected from up to eight of the soil borings described in Section 6.5.2. The anticipated locations of the groundwater grab samples are shown on Figure 8d. The final locations for the groundwater grab samples may be adjusted based on the results from sampling of existing monitoring wells. The final locations of groundwater grab samples will also be determined by the locations at which groundwater is encountered during drilling.

The final phase of groundwater investigation at former Building C-29 / the former East Fuel Farm will consist of installation of up to seven additional shallow monitoring wells. The locations of the shallow monitoring wells will be determined based on the results from the initial phases of the groundwater investigation described above. The proposed well locations will be provided to the County for review prior to installation.

The sampling and analysis plan for groundwater is outlined in Table 16. Groundwater grab samples from borings will be analyzed with an expedited turnaround time for decision-making regarding monitoring well installation. Groundwater sample analyses will include VOCs, DRO/ORO, GRO, dissolved MTCA metals, and natural attenuation parameters in accordance with Table 16. Groundwater samples from newly installed monitoring wells will be analyzed for VOCs. Additional analysis may be conducted based on the results of analysis of groundwater samples from existing monitoring wells and groundwater grab samples.

6.5.4 Soil Gas Investigation

Two soil gas samples (RISB-42 and RISB-48) will be collected from the suspected source areas to evaluate the potential for vapor intrusion for future buildings. Additional soil gas samples may be collected from selected locations where groundwater grab samples are planned (see Section 6.5.3), but where shallow groundwater is not encountered.

6.6 Deep Aquifer

This section identifies the data gaps, RI objectives, and planned RI activities for the deep aquifer.

6.6.1 Data Gaps and RI Objectives

The following data gaps were identified for the deep aquifer:

- Only three monitoring wells have been installed in the deep aquifer. Limited data are available regarding the direction of groundwater flow, including potential seasonal variations in flow. Flow direction has been determined to be predominantly to the north.
- Contaminant concentrations in the deep aquifer have not been evaluated since 2003.
- The extent of contamination in the aquifer has not been defined. Previous investigations associated with Building C-19 and former Building C-29 have identified TCE and its breakdown products (1,2-DCE, 1,2-DCA, and 1,2-DCP) in groundwater samples collected from the deep aquifer. The most recent sampling data for monitoring well DW1 (2003) indicate that concentrations of TCE in the aquifer were increasing.
- It is unknown if contamination identified in the deep aquifer at monitoring well DW2 is associated with the known release of TCE at Building C-19 or if it originates from a different source.

The objectives of the RI are to:

- Investigate the source or sources and delineate the extent of chlorinated VOCs in the deep aquifer.
- Evaluate groundwater flow conditions including direction of groundwater flow, flow rate, and other parameters.
- Evaluate natural attenuation parameters in the deep aquifer to assist in development of remedial alternatives.

6.6.2 Soil Investigation

The soil investigation will consist of collecting soil samples from soil borings advanced at the locations shown on Figure 9c, which will be submitted for laboratory analysis as described in Table 16. The explorations will consist of the following:

- One soil boring (RIDW-1) will be advanced in a presumed upgradient location to the south of Building C-19.
- One soil boring (RIDW-2) will be advanced in a presumed crossgradient location to the west of Building C-19 to further evaluate the direction of groundwater flow.
- One soil boring (RIDW-3) will be advanced in a presumed downgradient location to the north of Building C-19, between existing deep wells DW1 and DW2 to further delineate the extent of chlorinated VOCs.
- At each boring, up to five soil samples will be collected for laboratory analysis for VOCs as described in Table 16. The soil samples will be collected from the depth intervals where field screening indicates the likelihood for potential contamination, if any.
- Six soil samples will be selected from the above-described soil borings for analysis for total organic carbon and grain size.

- Up to three additional deep soil borings will be advanced at the site during a second mobilization. The locations of the soil borings will be determined based on the analytical results for the locations described above.

At each proposed exploration location, soil samples will be collected from borings advanced using a full-size rotosonic drilling rig. Each boring will be advanced to approximately 150 ft bgs to characterize the subsurface conditions. The soil sampling and analysis procedures are discussed in more detail in the SAP (Appendix A).

6.6.3 Groundwater Investigation

The groundwater investigation in this area will be completed in phases. Samples will be collected from existing monitoring wells prior to drilling new soil borings. In addition to the three existing monitoring wells, two groundwater grab samples will be collected from up to six additional deep soil borings described in Section 6.6.2. The anticipated locations of the first three deep borings and groundwater grab samples are shown on Figure 9c. The final locations for the groundwater grab samples may be adjusted based on the results from sampling of existing monitoring wells.

The final phase of groundwater investigation for the deep aquifer will consist of the installation of up to six additional deep monitoring wells. The locations of the first three deep monitoring wells are shown on Figure 9c. The locations of up to three additional deep monitoring wells will be determined based on the results from the initial phases of groundwater investigation described above. The proposed well locations will be provided to the County for review prior to installation.

The sampling and analysis plan for groundwater is outlined in Table 16. Groundwater grab samples from the well borings will be analyzed with an expedited turnaround time for decision-making regarding additional boring/monitoring well installation. Groundwater sample analyses will include VOCs, 1,4-dioxane, and natural attenuation parameters in accordance with Table 16. Groundwater samples from newly installed monitoring wells will be analyzed for VOCs. Additional analysis may be conducted based on the results of analysis of groundwater samples from existing monitoring wells and groundwater grab samples.

6.7 General Procedures for Groundwater Investigation

The following general procedures will be used for the groundwater investigation at the Site. Additional details are provided in the SAP (Appendix A).

6.7.1 Monitoring Well Installation

Rotosonic drilling methods will be used to install the monitoring wells, which will be constructed with 2-inch-diameter PVC casings and well screens. Well construction and installation is discussed in more detail in the SAP (Appendix A).

During drilling, soil samples will be collected for inspection and geologic classification, and to document the shallow subsurface stratigraphy. The geologic information will be used to develop cross sections and validate the Conceptual Site Model that will be presented in the RI report.

The proposed locations for the monitoring wells are shown on Figures 5d, 6d, 7e, 8d, and 9c. The locations were selected to provide spatial coverage across the Site, and to evaluate groundwater conditions upgradient and downgradient of the identified investigation areas. Additional monitoring wells may be added, or the locations of the monitoring wells may be adjusted, as warranted, based on the findings of the initial phase of the groundwater investigation and discussions with the County.

6.7.2 Frequency of Sampling

Groundwater elevations will be monitored seasonally (one wet season and one dry season) for 1 year to evaluate the groundwater flow direction and gradient. Two rounds of groundwater samples will be collected for laboratory analysis.

6.7.3 Laboratory Analysis

The groundwater samples collected from monitoring wells will be submitted for laboratory analysis as described in the preceding sections. Methods for the laboratory analyses are described in the SAP (Appendix A).

6.7.4 Groundwater Flow Monitoring

To evaluate seasonal groundwater flow direction, the depth to groundwater will be measured from a surveyed reference point at each well casing and the depths converted to elevations for one wet season and one dry season monitoring event for both shallow monitoring wells and aquifer monitoring wells. Elevations will be plotted maps and contoured. Detailed procedures for monitoring groundwater elevations are provided in the SAP (Appendix A).

6.8 Site Utility and Tank Investigation

This section discusses identifying and confirming the locations of existing utilities at the Site.

6.8.1 Utility Survey

Historical documentation has been reviewed and compiled to create Figure 10, which shows the approximate location of all known utilities at the Site. Additional, more expansive, utility survey work may be necessary if RI analytical results and field observations indicate that a utility might be acting as a preferential pathway. This more detailed utility survey could include a private utility locating company employing magnetometers, radio frequency, ground-penetrating radar (GPR), and utility cameras, as necessary.

6.8.2 Underground Storage Tank Reconnaissance

Following review of historical documentation, additional information is necessary related to the status of the following current and former USTs:

- C-22: Confirm removal of USTs off the north end of Building C-22
- East Fuel Farm: Determine how many USTs still exist at the former East Fuel Farm.
- C-23: Evaluate potential USTs at the southwest corner of Building C-23.

As with the potential need for an expanded utility survey noted above, a UST reconnaissance may include one or more of the following exploratory methods: metal detection using a magnetometer, GPR, and air knife/vacuum exploration.

7.0 FEASIBILITY STUDY

The purpose of the FS is to develop, evaluate, and select cleanup action alternatives for the Site. The FS will:

- Identify applicable or relevant and appropriate requirements (ARARs) for Site cleanup
- Identify media and locations where remedial action is needed
- Develop remedial action objectives (RAOs)
- Develop, screen, and evaluate cleanup alternatives
- Identify a preferred alternative.

The following sections provide additional discussion of details for each of the above bulleted items.

7.1 Applicable or Relevant and Appropriate Requirements

In accordance with MTCA, all cleanup actions must comply with applicable state and federal laws [WAC 173-340-710(1)]. MTCA defines applicable state and federal laws to include legally applicable requirements and those requirements that are relevant and appropriate. Collectively, these requirements are referred to as ARARs. The starting point for ARARs is the MTCA cleanup levels and regulations that address implementation of a cleanup under MTCA (Chapter 173.105D of the Revised Code of Washington [RCW]; Chapter 173-340 WAC). Other potential ARARs may include the following:

- Washington State Sediment Management Standards (Chapter 173-204 WAC)
- State Water Pollution Control Act (Chapter 90.48 RCW)
- US Environmental Protection Agency (EPA) National Recommended Water Quality Criteria – Section 304 Clean Water Act
- EPA Water Quality Standards (National Toxics Rule) – 40 Code of Federal Regulations (CFR) 131
- Minimum Standards for Construction and Maintenance of Wells (Chapter 173-160 RCW)
- Washington Pollution Control Act and the implementing regulations, Water Quality Standards for Surface Waters of the State of Washington (Chapter 173-201A WAC)
- Washington Hazardous Waste Management Act and the implementing regulations, Dangerous Waste Regulations (Chapter 173-303 WAC), to the extent that any dangerous wastes are discovered or generated during the cleanup action
- The Federal Clean Water Act, with respect to in-water work associated with dredging or sediment capping
- Washington's Shoreline Management Act, with respect to construction activities conducted near the shoreline during the cleanup action
- Endangered Species Act, due to listing of Puget Sound chinook salmon and the potential listing of coastal/Puget Sound bull trout
- Washington Clean Air Act (Chapter 70.94 WAC)

- Occupational Safety and Health Act, 29 CFR Subpart 1910.120
- Washington Industrial Safety and Health Act.

From MTCA and the other ARARs, cleanup levels and points of compliance (collectively referred to as cleanup standards) will be developed for media and parameters that require remedial action. In addition, the FS will identify likely permits required for implementation of the cleanup action.

7.2 Delineation of Media Requiring Remedial Action

The RI process will determine if soil or groundwater analytical results exceed cleanup levels and, if so, identify the locations of the exceedances. Based on any exceedances and the established points of compliance, the FS will identify the areas that require remedial action.

7.3 Development of Remedial Action Objectives

The RAOs identify the goals that must be achieved by a cleanup alternative in order to meet cleanup standards and provide adequate protection of human health and environment. The RAOs must address all affected media and a cleanup alternative must achieve all RAOs to be considered a viable cleanup action. RAOs will be developed for portions of the Site requiring remedial action.

The RAOs will be action-specific and/or media-specific. Action-specific RAOs are based on actions required for environmental protection that are not intended to achieve a specific chemical criterion. Media-specific RAOs are based on the cleanup levels. The RAOs will specify the COCs, the potential exposure pathways and receptors, and acceptable contaminant levels or range of levels for each exposure pathway, as appropriate.

The extent to which each alternative meets the RAOs will be determined by applying the specific evaluation criteria identified in the MTCA regulation.

7.4 Screening of Cleanup Alternatives

Cleanup alternatives will be developed for portions of the Site that require remedial action. Initially, general remediation technologies will be identified for the purpose of meeting RAOs. General remediation technologies consist of specific remedial action technologies and process options. General remediation technologies will be considered and evaluated based on the properties of identified contaminant(s) and may include institutional controls, containment, or other engineering controls, removal, *in situ* treatment, and natural attenuation.

Specific remedial action technologies are the engineering components of a general remediation technology and process options are those specific processes within each specific technology. Specific remedial action technologies and representative process options will be selected for evaluation based on documented development or documented successful use for the observed contamination conditions at the Site. Cleanup alternatives will be developed from the general and specific remedial technologies and process options consistent with Ecology's expectations identified in WAC 173-340-370 using best professional judgment and guidance documents as appropriate [e.g., Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (EPA 1988)].

During the development of cleanup alternatives, both the current and planned future land use will be considered.

7.5 Evaluation of Cleanup Alternatives

MTCA requires that cleanup alternatives be compared to a number of criteria as set forth in WAC 173-340-360 to evaluate the adequacy of each alternative in achieving the intent of the regulations, and as a basis for comparing the relative merits of the developed cleanup alternatives. Consistent with MTCA, the alternatives will be evaluated with respect to compliance with threshold requirements, permanence, and restoration timeframe; the results of the evaluation will be documented in the RI/FS reports.

7.5.1 Threshold Requirements

As specified in WAC 173-340-360(2)(a), all cleanup actions are required to meet the following threshold requirements:

- Protection of human health and the environment
- Compliance with cleanup standards specified under MTCA
- Compliance with applicable state and federal laws
- Provisions for compliance monitoring.

7.5.2 Requirement for Permanent Solution to the Maximum Extent Practicable

WAC 173-340-200 defines a permanent solution as one in which cleanup standards can be met without further action being required at the original site or any other site involved with the cleanup action, other than the approved disposal site for any residue from the treatment of hazardous substances. Ecology recognizes that permanent solutions may not be practicable for all sites. To determine whether a cleanup action is permanent to the "maximum extent practicable," MTCA requires that a disproportionate cost analysis [WAC 173-340-360(3)(b)] be used. In accordance with WAC 173 340-360(3)(f), the following criteria will be used to evaluate and compare each cleanup action alternative when conducting a disproportionate cost analysis:

- Overall protectiveness of human health and the environment, including the degree to which Site risks are reduced, the risks during implementation, and the improvement of overall environmental quality
- Long-term effectiveness, including the degree of certainty that the alternative will be successful, the long-term reliability, the magnitude of residual risk, and the effectiveness of controls required to manage treatment residues and remaining waste

- Management of short-term risks, including the protection of human health and the environment during construction and implementation
- Permanent reduction in toxicity, mobility, and volume of hazardous substances, including the reduction or elimination of hazardous substance releases and sources of releases
- Implementability, including consideration of whether the alternative is technically possible; the availability of necessary offsite facilities, services, and materials; administrative and regulatory requirements; scheduling, size, and complexity of construction; monitoring requirements; access for construction, operations, and monitoring; and integration with existing facility operations
- Cleanup costs, including capital costs and operation and maintenance costs
- Consideration of public concerns, which will be addressed through public comment on the cleanup action plan.

Procedures that will be used for conducting a disproportionate cost analysis are described in Section 7.6.

7.5.3 Requirements for a Reasonable Restoration Timeframe

WAC 173-340-360(4)(b) specifies that the following factors be considered in establishing a “reasonable” timeframe:

- Potential risks to human health and the environment
- Practicability of achieving a shorter restoration timeframe
- Current use of the site, surrounding areas, and associated resources that are, or may be, affected by releases from the site
- Potential future use of the site, surrounding areas, and associated resources that are, or may be, affected by releases from the site
- Availability of alternate water supplies
- Likely effectiveness and reliability of institutional controls
- Ability to control and monitor migration of hazardous substances from the site
- Toxicity of the hazardous substances at the site
- Natural processes that reduce concentrations of hazardous substances and have been documented to occur at the site or under similar site conditions.

7.6 Disproportionate Cost Analysis Procedures

As described in Section 7.5.2, MTCA requires that cleanup actions be permanent to the maximum extent practicable and requires that a disproportionate cost analysis be used when the cleanup alternatives being considered are not permanent as defined under WAC 173-340-200. Evaluation of the practicability of a given alternative is a comparative evaluation of whether the incremental increase in cost associated with increasingly protective cleanup actions is substantial and disproportionate to the incremental increase in environmental benefit. In the disproportionate cost analysis, cleanup alternatives are arranged from most to least permanent based on the criteria specified in WAC 173-340-360(f) and described in Section 7.5.2. Costs are disproportionate to benefits if the incremental costs of the more permanent alternative exceed the incremental benefits achieved by the lower-cost alternative [WAC 173-340-360(3)(e)(i)]. Alternatives that exhibit disproportionate costs are considered “impracticable.” Where the benefits of two alternatives are equivalent, MTCA specifies that Ecology select the least costly alternative [WAC 173-340-360(e)(ii)(C)].

7.7 Recommendation of Remedial Action Alternatives

This section of the FS will recommend a remedial action alternative based on the results of the comparative evaluation. The recommended alternative will meet the minimum requirements for cleanup actions: protect human health and the environment, comply with cleanup standards, comply with applicable state and federal laws, provide for compliance monitoring, use permanent solutions to the extent practicable, provide for a reasonable timeframe, and consider public concerns.

8.0 REPORTING

An RI report will be prepared in accordance with MTCA (WAC 173-340-350) to document the investigative activities conducted at the Site and the nature and extent of contamination, and provide the data required to support the preparation of an FS for potential cleanup actions at the Site. The RI report will document the findings of the activities outlined in this work plan and the data collected using text, tables, figures, geologic/hydrogeologic cross sections, and groundwater contour maps, as appropriate. The report will include an assessment of the quality of the analytical data based on a quality assurance/quality control review, and present and discuss the data relative to the screening levels and the MTCA cleanup levels.

The RI report will incorporate, as appropriate, the findings of the RI field investigations with existing data from previous investigations and remedial actions at the Site to document and evaluate the Conceptual Site Model including: the Site stratigraphy, hydrogeology, seasonal groundwater occurrence and flow, potential receptors and pathways, and potential contaminant occurrence and migration.

The FS report will be prepared as described in Section 7.0 of this work plan and in accordance with WAC 173-340-350.

Project progress reports will be submitted to the County monthly during the RI phase of the project and will include a description of activities completed in the prior month, summaries of significant findings, deviations from the approved work plan, summaries of problems or anticipated problems with meeting schedules and objectives, solutions developed and implemented to address actual or anticipated problems or delays, changes in key personnel, and a description of work planned for the next reporting period.

9.0 SCHEDULE

A preliminary schedule for the RI/FS is summarized below. This schedule assumes that all necessary approvals of this work plan will be in place by October 15, 2018.

Tasks	Date Range
Repair and redevelopment of existing monitoring wells, utility and UST locating, and collection and laboratory analysis of groundwater samples from existing wells	10/15/2018 – 12/15/2018
Collection and laboratory analysis of soil, groundwater grab, and soil gas samples from new soil borings in each of the investigation areas and installation and sampling of three new deep monitoring wells	12/15/2018 – 05/01/2019
Installation and sampling of shallow monitoring wells in each investigation area based on the analytical results for groundwater samples collected from existing monitoring wells and the analytical results for the groundwater grab samples	03/01/2019 – 09/01/2019
Installation of additional deep monitoring wells and collection and analysis of supplemental soil, groundwater, and soil gas samples to fill data gaps remaining after previous phases of investigation	05/01/2019 – 09/01/2019
Completion of a second round of groundwater monitoring from existing and newly installed monitoring wells to evaluate seasonal variations in groundwater conditions	08/01/2019 – 10/01/2019
Preparation of RI report	05/01/2019 – 12/31/2019
Preparation of FS report	01/01/2020 – 06/01/2020

Variations from this schedule may be necessary based on unanticipated findings, Site access constraints, weather delays, and potential revisions to the existing scope and budget authorization, if needed. If additional RI activities are needed to meet the objectives of the RI work plan, the scope, schedule, and submittal requirements for this additional work will be developed and submitted to the County for review and concurrence.

10.0 USE OF THIS WORK PLAN

This work plan has been prepared for the use of Snohomish County and its designated representatives for specific application to the remedial investigation at the TECT Aerospace Lease Area at Paine Field in Everett, Washington. No other party, except applicable regulatory agencies, is entitled to rely on the information, conclusions, and recommendations included in this document without the express written consent of LAI. Further, the reuse of information and recommendations provided herein for extensions of this project or for any other project, without review and authorization by LAI, shall be at the user’s sole risk. LAI warrants that within the limitations of scope, schedule, and budget, our services have been provided in a manner consistent with that level of care and skill ordinarily exercised by members of the profession currently practicing in the same locality under similar conditions as this project. We make no other warranty, either express or implied.

11.0 REFERENCES

AGI. 1993. Value Engineering Assessment, East and West Fuel Farms and "Old Mike" Soil Stockpile, Snohomish County Airport, Everett, Washington. Applied Geotechnology Inc. November 24.

AGI. 1994. Final Report: Contamination Assessment, Paine Field Fuel FArms and Old Mike Soil Stockpile, Snohomish County Airport, Snohomish County, Washington. AGI Technologies. August 12.

AGI. 1999. Report: Preliminary Contamination Assessment, All Fab (Former), Snohomish County Airport, Paine Field, Everett, Washington. AGI Technologies. June 23.

Booth, D.B., K.G. Troost, J.J. Clague, and R.B. Waitt. 2004. "The Cordilleran Ice Sheet." In The Quarternary Period in the United States, edited by A. Gillespie, S.C. Porter, and B. Atwater, pp. 17-43. Amsterdam, The Netherlands: Elsevier Press.

CDM. 2000a. Aquifer Investigation, All Fab (Former), Snohomish County Airport, Everett, Washington. Camp Dresser & McKee Inc. December 12.

CDM. 2000b. Underground Storage Tank Closure Assessment, East and West Fuel Farms, Paine Field, Everett, Washington. Camp Dresser & McKee Inc. October 17.

CDM. 2002. Hydrocarbon Evaluation, East and West Fuel Farms, Snohomish County Airport, Snohomish County, Washington. Camp Dresser & McKee Inc. February 26.

CDM. 2005. Former All Fab, Building C-19 Chlorinated Solvent Delineation, Snohomish County Airport, Everett, Washington. Camp Dresser & McKee Inc. February 14.

Ecology. 1994. Natural Background Soil Metals Concentrations in Washington State. Publication No. 94-115. Washington State Department of Ecology. October. https://fortress.wa.gov/ecy/publications/documents/94115.pdf.

Ecology. 2013. Letter: No Further Action at the Following Site: Site Name: Paine Field C-19 Giddens (AKA Tect Aerospace Everett 2011); Site Address: 11002 29th Avenue West, Building C-19 Paine Field, Everett, Washington; Facility/Site No.: 2851; VCP Project No.: NW2554; Cleanup Site No.: 3808. From Roger K. Nye, Toxics Cleanup Program, Washington State Department of Ecology, to Timothy Giddens. May 22.

Ecology. 2015. "Cleanup Levels and Risk Calculation (CLARC)." Washington State Department of Ecology. https://fortress.wa.gov/ecy/clarc/CLARCHome.aspx.

Envirotech. 1997. Phase I Environmental Site Assessment, Building C-19, Paine Field, Everett, Washington. Envirotech Systems, Inc. October.

EPA. 1988. Interim Final: Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA. EPA/540/G-89/004. US Environmental Protection Agency. October.

EPI. 2006. Site Characterization Letter Report, Building C-19, Paine Field, 11002 29th Avenue West, Everett, Washington 98204. March 17.

EPI. 2007. Additional Site Characterization Letter Report, Building C-19, Paine Field, 11002 29th Avenue West, Everett, Washington 98204. Environmental Partners Inc. April 5.

EPI. 2010. November 2010 Site Characterization Letter Report, Building C-19, Paine Field, 11002 29th Avenue West, Everett, Washington 98204. Environmental Partners Inc. December 6.

EPI. 2011. Remedial Action Report - Bay 3, Building C-19, Payne (sic) Field, 1102 (sic) 29th Avenue West, Everett, Washington 98204. Environmental Partners Inc. April 8.

EPI. 2012. Remedial Action Report - Bays 1 and 2, Building C-19, Paine Field, 1102 (sic) 29th Avenue West, Everett, Washington 98204. Environmental Partners Inc. January 6.

LAI. 1993. Report: Phase I Environmental Site Assessment, All Fab, Inc. - Paine Field, Everett, Washington. Landau Associates, Inc. November 18.

LAI. 1994. Report: Degreaser Pit Investigation, Building C-19, All Fab, Inc. - Paine Field, Everett, Washington. Landau Associates, Inc. April 25.

LAI. 1995. Report: Environmental Sampling and Analysis, Building C-19 Sump, Snohomish County Airport. Landau Associates, Inc. August 15.

LAI. 1996. Interim Environmental Investigation Results Summary, Former All Fab, Inc. Chemical Storage Building (Building C-29), Snohomish County Airport. Landau Associates, Inc. July 24.

LAI. 2017. Report: Phase I Environmental Site Assessment, TECT Aerospace Lease Area, Paine Field/Snohomish County Airport, Snohomish County, Washington. Landau Associates, Inc. May 16.

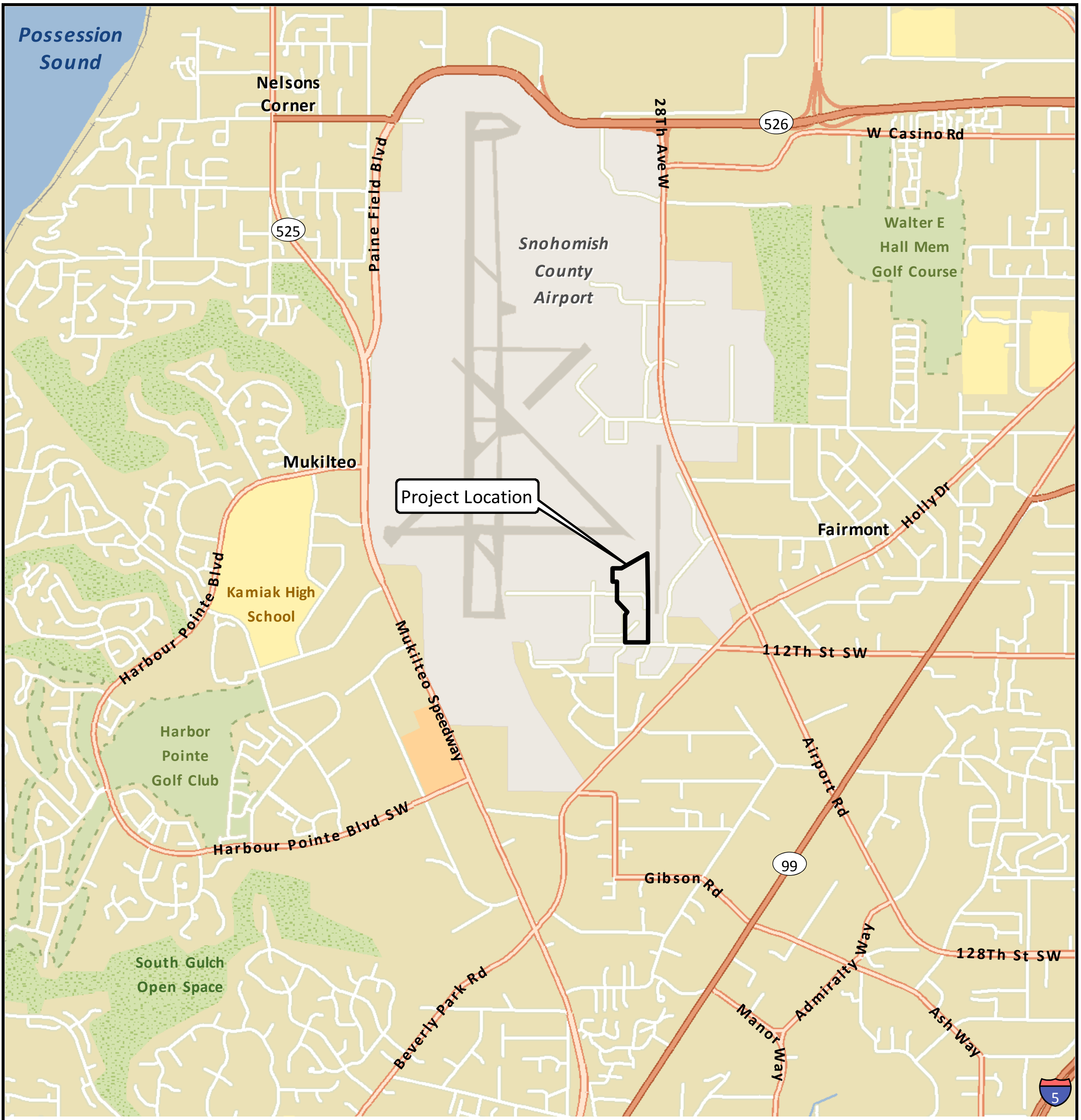
LAI. 2018. Technical Memorandum: Focused Phase II Environmental Site Assessment Data Summary, TECT Aerospace, Everett, Washington. Landau Associates, Inc. February 8.

Neil, T. 2017. "Re: Paine Field Supplemental Investigation." From Terry Neil, Maintenance/EHS Manager, TECT Aerospace, to Kathryn Hartley, Associate, Landau Associates, Inc. June 15.

Rardin, A. 2017. "Re: Subject Property History." Andrew Rardin, Environmental and Wildlife Manager, Paine Field/Snohomish County Airport. April 26.

Snohomish County. "Permitting and Land Use Maps." https://snohomishcountywa.gov/2467/Permitting-and-Land-Use-Maps.

Snohomish County Airport. 1996. Letter: Greenish-Yellow Tinted Water at the Foundation of Building C-29. From Andrew Rardin, to Toxics Cleanup Program, Washington State Department of Ecology. January 22.



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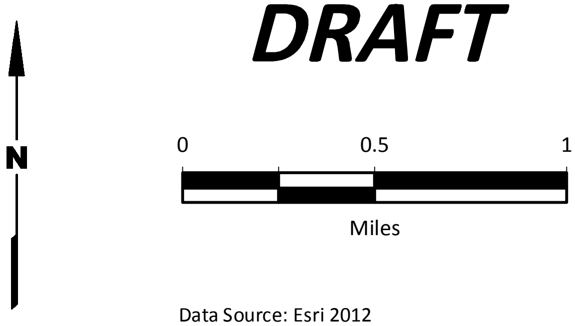

Data Source: Esri 2012

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Everett, Washington

Vicinity Map

Figure
1

G:\Projects\222\057\010\013\RI Work Plan\F02SitePlan.mxd 9/17/2018

Legend

▭ Approximate Site Boundary

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 150 300

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

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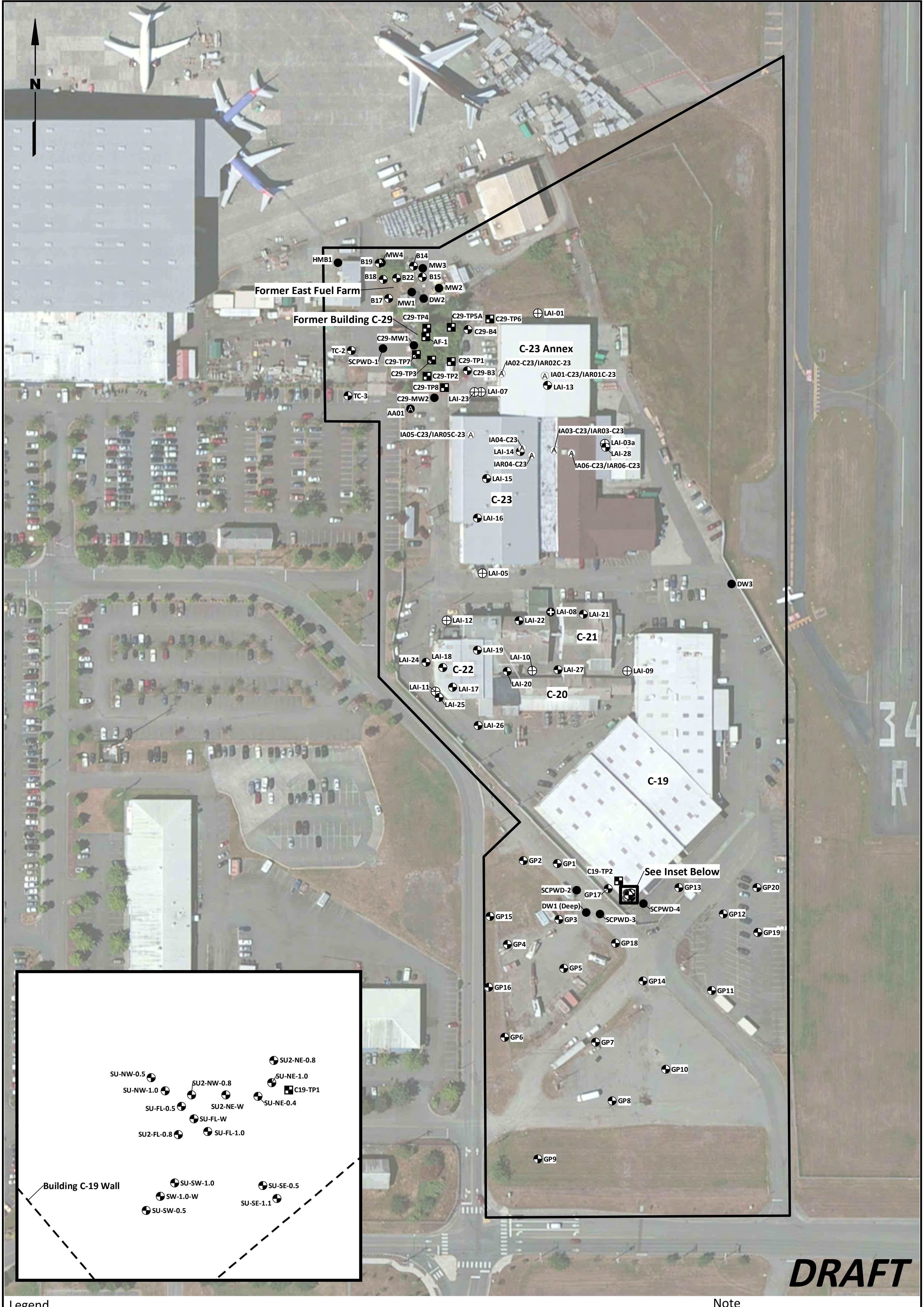
TECT Aerospace Everett, Washington	**Site Plan**	Figure 2

G:\Projects\222\057\010\013\RI Work Plan\F03SampleLocations.mxd 9/17/2018



Legend

- ● Ambient Air Sampling Location
- ● Soil Boring
- Ⓐ Indoor Air Sampling Location
- ● Monitoring Well
- ⊕ Soil/Soil Gas Sampling Location
- ⊕ Soil Gas Sampling Location
- ▨ Test Pit
- ▭ Approximate Site Boundary

0 125 250

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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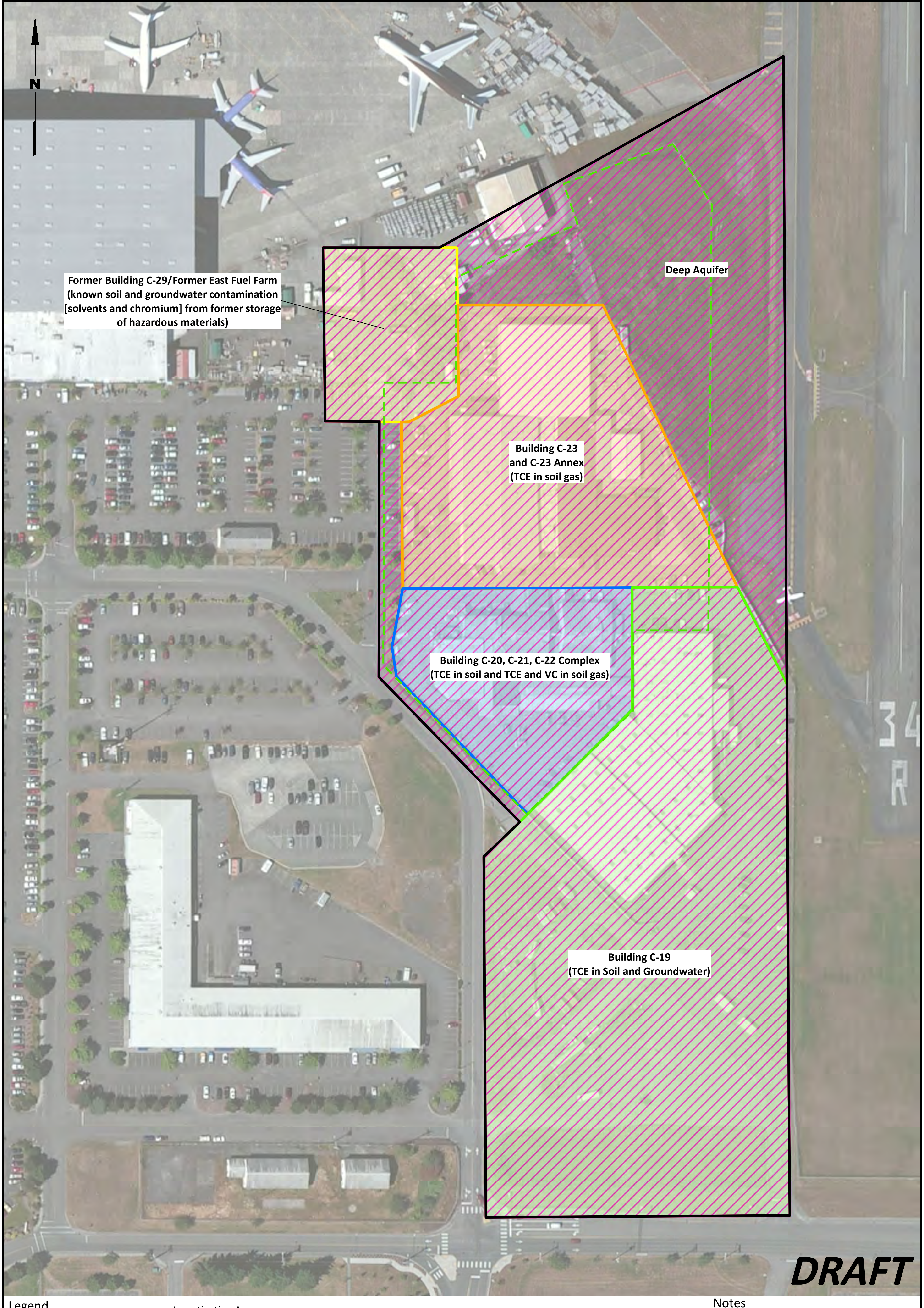
Everett, Washington

Pre-RI Investigation Locations

Figure
3

G:\Projects\222\057\010\013\RI Work Plan\F04InvestigationAreas.mxd 9/17/2018



Legend

□ Approximate Site Boundary

⬚ Current TECT Lease Area

Investigation Areas

▨ Deep Aquifer

🟩 Building C-19

🟦 Building C-20, C-21, C-22 Complex

🟧 Building C-23 and C-23 Annex

🟨 Former Building C-29/ Former East Fuel Farm

0 125 250

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

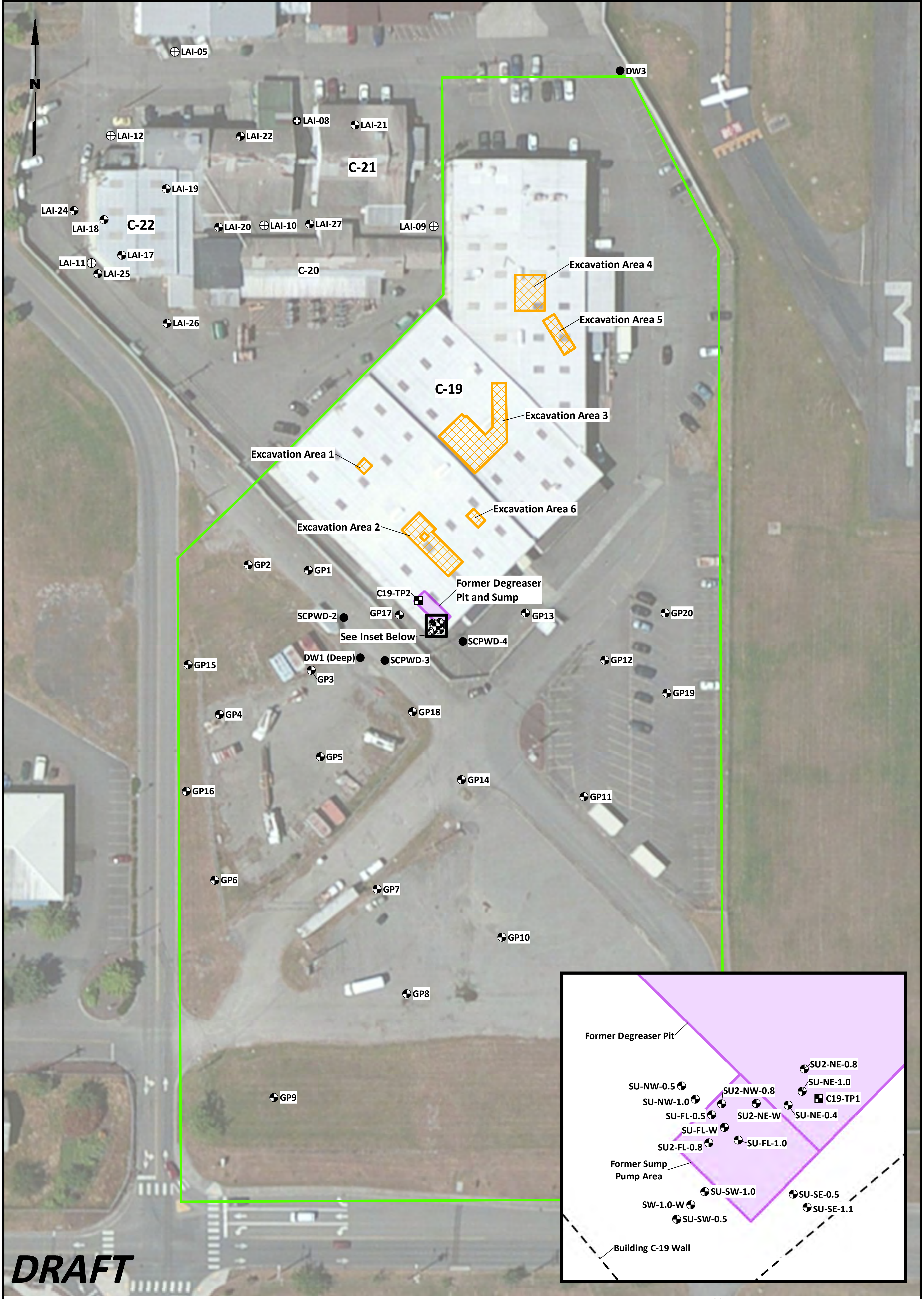
Notes

1. TCE = trichloroethene
   VC = vinyl chloride
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

LANDAU ASSOCIATES

TECT Aerospace Everett, Washington	**Investigation Areas**	Figure 4

G:\Projects\222\057\010\013\RI Work Plan\F05aBuildingC-19AOCPreRIExplorations.mxd 9/17/2018



Legend

- ⊕ Soil Boring
- Ⓐ Indoor Air Sampling Location
- ● Monitoring Well
- ⊕ Soil/Soil Gas Sampling Location
- ⊕ Soil Gas Sampling
- ▣ Test Pit
- 🟩 Building C-19 Investigation Area
- ▨ Remedial Action Areas

0 70 140

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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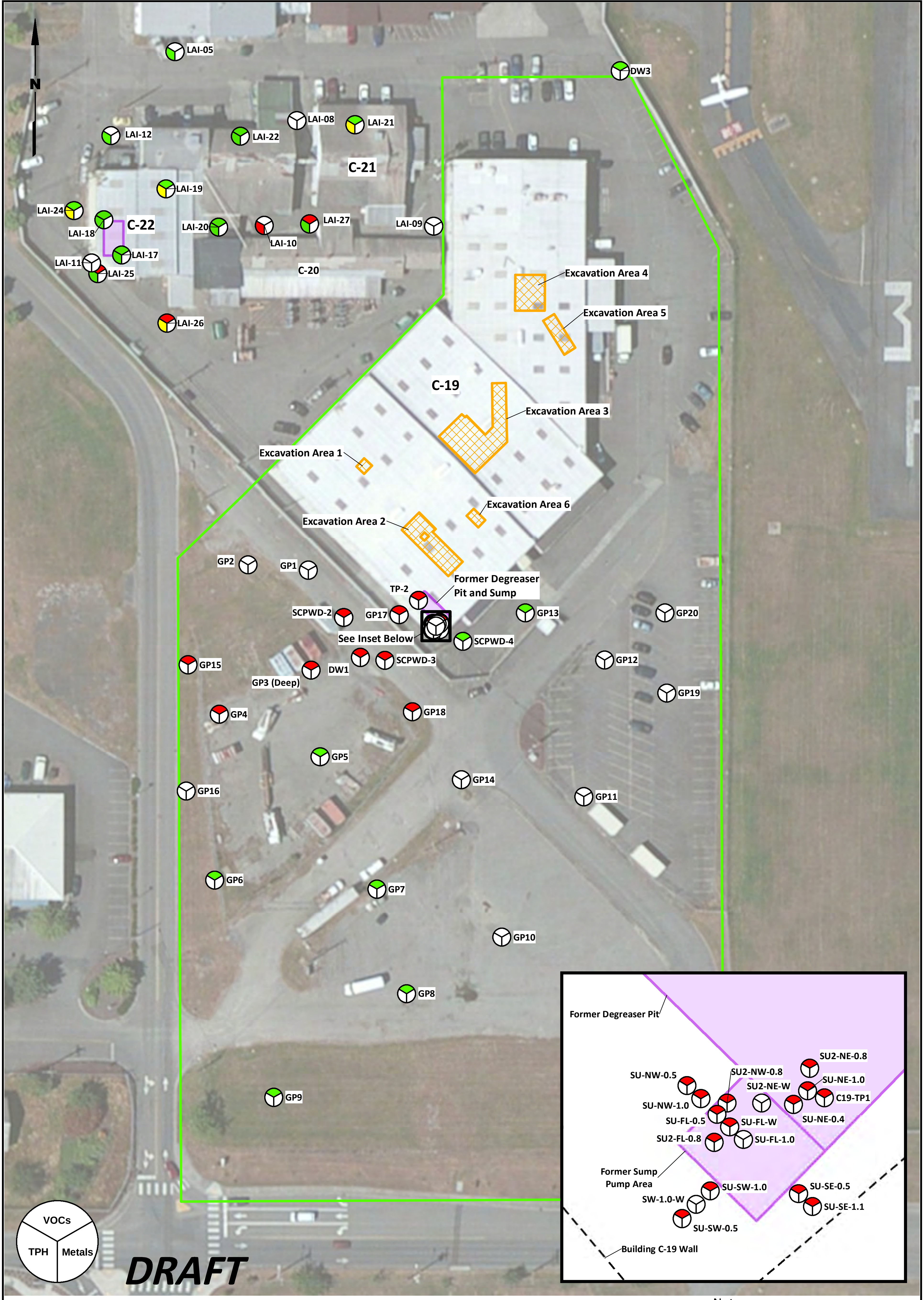
TECT Aerospace
Everett, Washington

Building C-19
Pre-RI Exploration Locations

Figure
5a

G:\Projects\222\057\010\013\RI Work Plan\F05bBuildingC-19AOCPreRISoilExceedances.mxd 9/17/2018



Legend

○ Sampling Location

🟩 Building C-19 Investigation Area

▨ Remedial Action Areas

◔ Not Analyzed

◔ Analyzed, Detected, No Exceedance

◔ Analyzed, Not Detected

◔ Exceedance

0 70 140

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes

1. TPH = total petroleum hydrocarbons
VOCs = volatile organic compounds
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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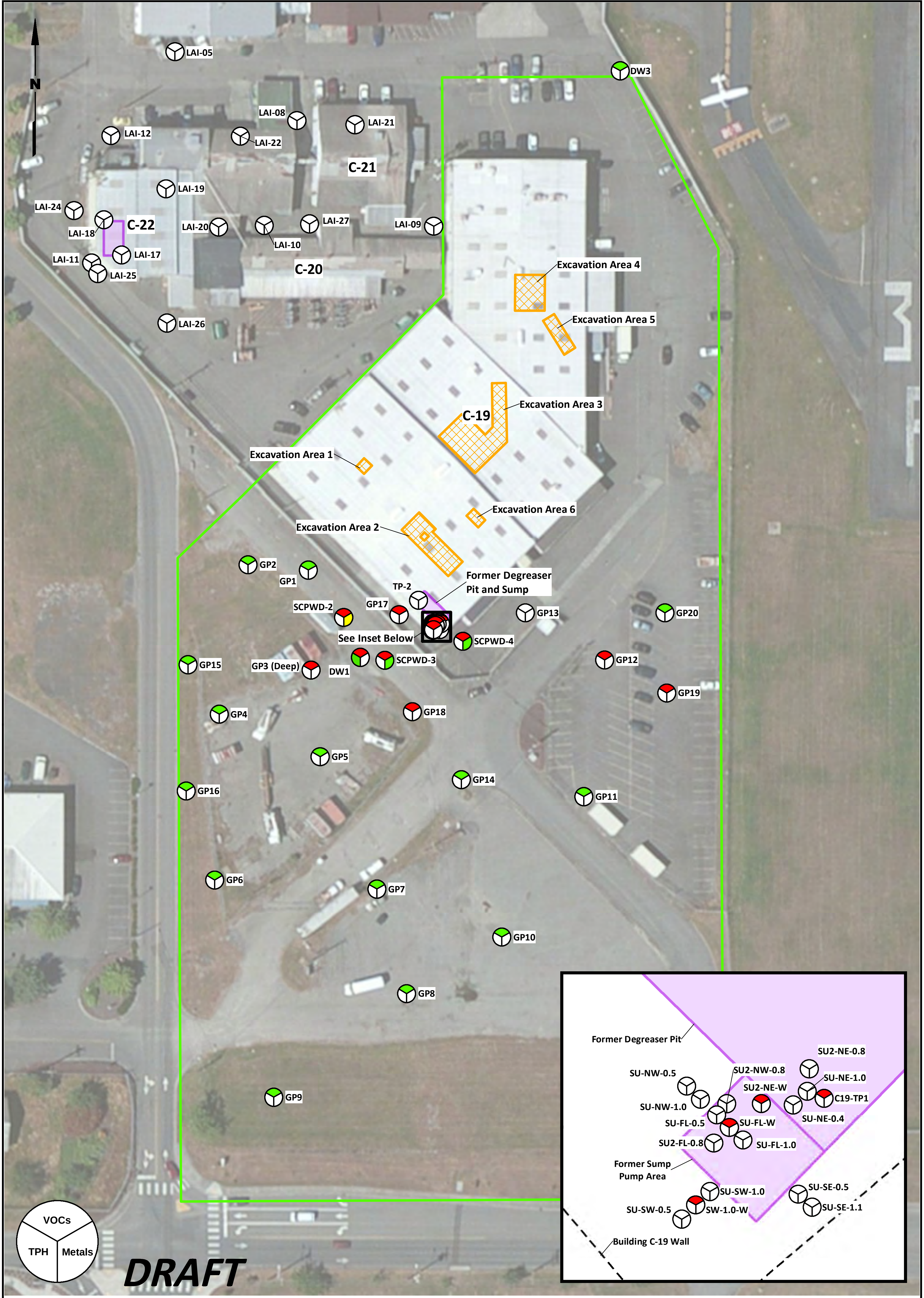
Everett, Washington

Building C-19
Pre-RI Soil Results

Figure
5b

G:\Projects\222\057\010\013\RI Work Plan\F05cBuildingC-19AOCPreRIGWExceedances.mxd 9/17/2018



Legend

- Sampling Location
- 🟩 Building C-19 Investigation Area
- ▨ Remedial Action Areas
- Not Analyzed
- Analyzed, Detected, No Exceedance
- Analyzed, Not Detected
- Exceedance

0 70 140

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes

1. TPH = total petroleum hydrocarbons
   VOCs = volatile organic compounds
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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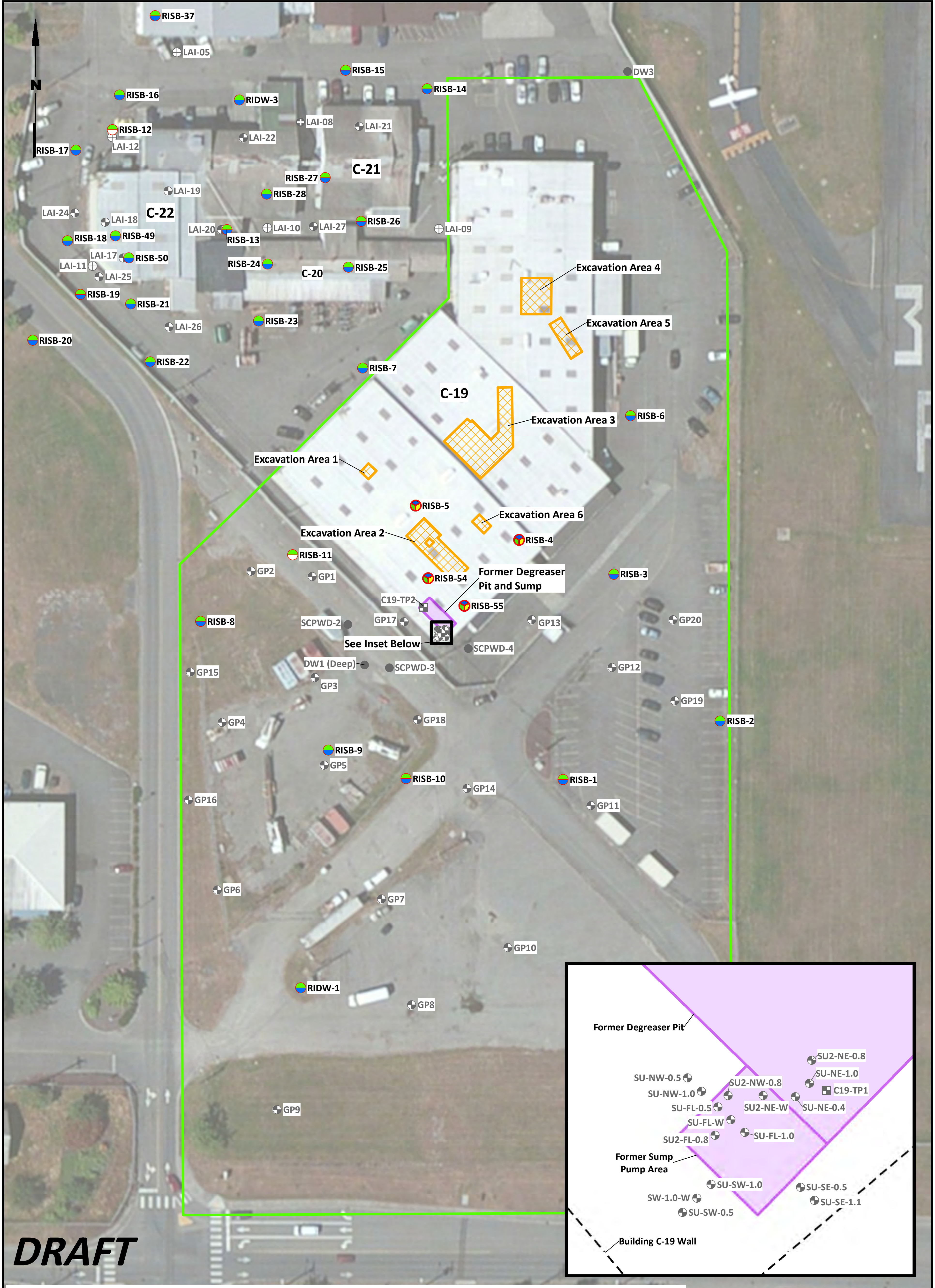
Building C-19
Pre-RI Groundwater Results

Figure
5c

G:\Projects\222\057\010\013\RI Work Plan\F05dBuildingC-19AOCProposedRIExplorations.mxd 9/17/2018



Legend

- Proposed Soil Sampling Location
- Proposed Soil and Groundwater Sampling Location
- Proposed Soil, Soil Gas, and Groundwater Sampling Location
- Soil Boring
- Indoor Air Sampling Location
- ● Monitoring Well
- Soil/Soil Gas Sampling Location
- Soil Gas Sampling Location
- Test Pit
- 🟩 Building C-19 Investigation Area
- ▨ Remedial Action Areas

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

0 70 140

Scale in Feet

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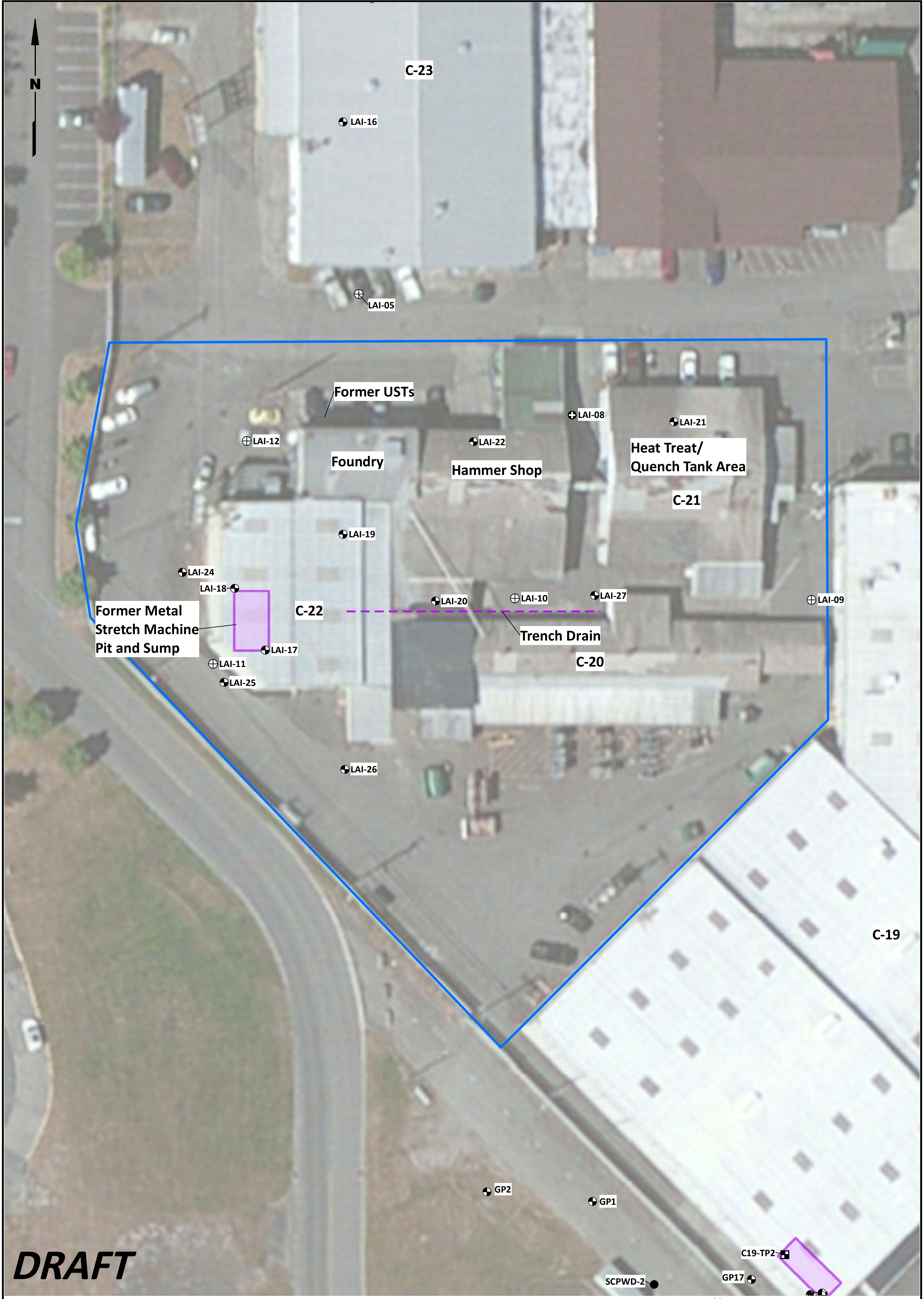
Building C-19
Proposed RI Exploration Locations

Figure
5d

G:\Projects\222\057\010\013\RI Work Plan\F06aBuildingC-20C-21C22ComplexAOCPreRIExplorations.mxd 9/17/2018



Legend

◕ Soil Boring

● Monitoring Well

⊕ Soil/Soil Gas Sampling Location

✚ Soil Gas Sampling Location

▣ Test Pit

▭ Building C-20, C-21, C-22 Complex Investigation Area

0 40 80

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes

1. UST = underground storage tank.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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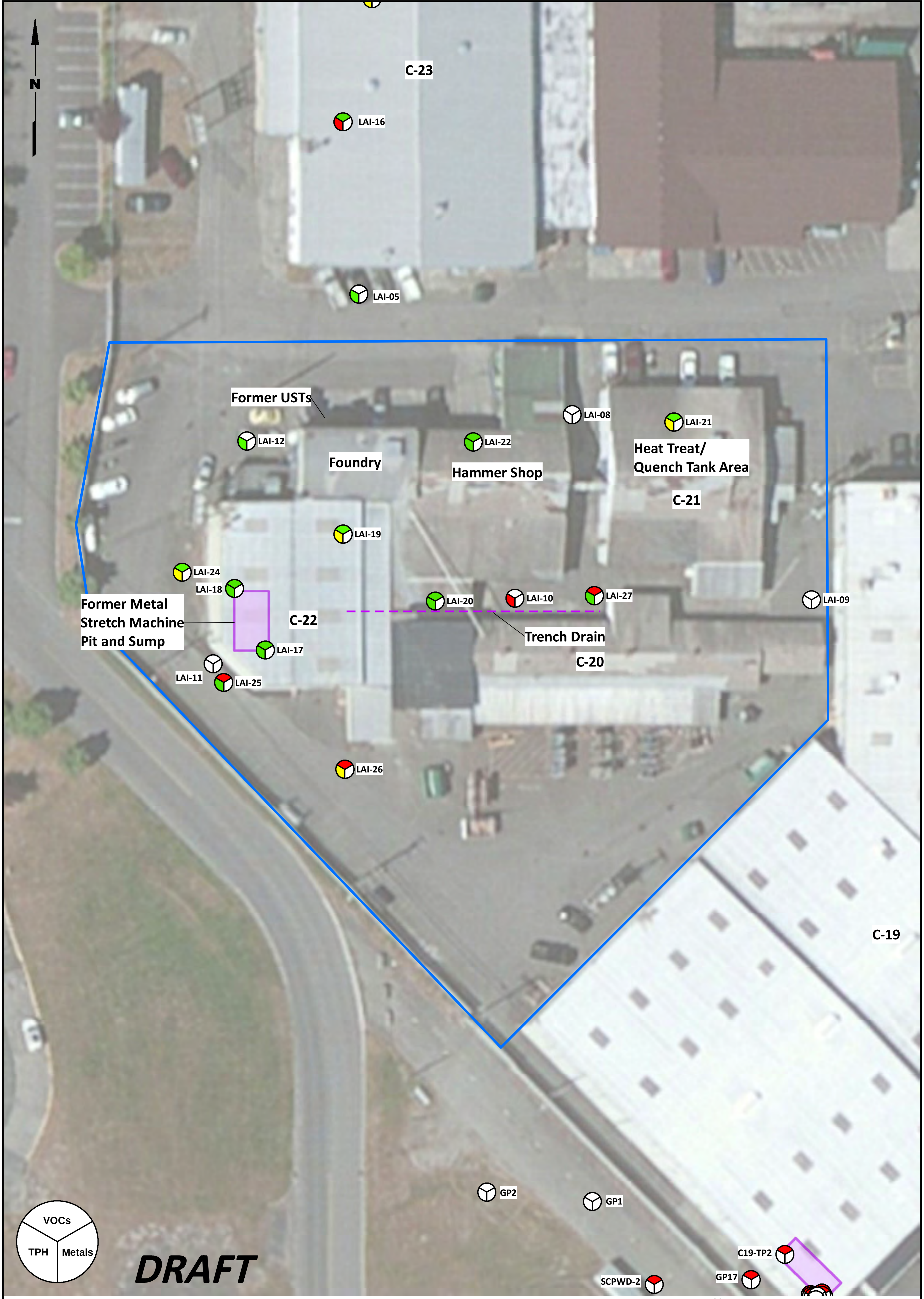
Everett, Washington

Building C-20, C21, C22 Complex
Pre-RI Exploration Locations

Figure
6a

G:\Projects\222\057\010\013\RI Work Plan\F06bBuildingC-20C-21C22ComplexAOCPreRISoilExceedances.mxd 9/17/2018



Legend

Sampling Location

Building C-20, C-21, C-22 Complex Investigation Area

Not Analyzed

Analyzed, Detected, No Exceedance

Analyzed, Not Detected

Exceedance

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes
1. TPH = total petroleum hydrocarbons
VOCs = volatile organic compounds.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 40 80
Scale in Feet

LANDAU ASSOCIATES

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Everett, Washington

Building C-20, C-21, C-22 Complex
Pre-RI Soil Results

Figure
6b

G:\Projects\222\057\010\013\RI Work Plan\F06cBuildingC-20C-21C22ComplexAOCPreRISoilGasExceedances.mxd 9/17/2018

Legend

○ Soil Gas Not Analyzed

🟢 Soil Gas Analyzed, Not Detected

🟡 Soil Gas Analyzed, Detected, No Exceedance

🔴 Soil Gas Exceedance

▭ Building C-20, C-21, C-22 Complex Investigation Area

0 40 80

Scale in Feet

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Building C-20, C-21, C-22 Complex
Pre-RI Soil Gas Results

Figure
6c

G:\Projects\222\057\010\013\RI Work Plan\F06dBuildingC-20C-21C22ComplexAOCProposedRIExplorations.mxd 9/17/2018

Legend

- Proposed Soil Sampling Location
- Proposed Soil and Groundwater Sampling Location
- Soil, Soil Gas, and Groundwater Sampling Location
- Soil Boring
- ● Monitoring Well
- ⊕ Soil/Soil Gas Sampling Location
- Soil Gas Sampling Location
- Test Pit
- Building C-20, C-21, C-22 Complex Investigation Area

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

0 40 80

Scale in Feet

Landau Associates

TECT Aerospace
Everett, Washington

Building C-20, C-21, C-22 Complex
Proposed RI Exploration Locations

Figure
6d

G:\Projects\222\057\010\013\RI Work Plan\F07aBuildingC23AOCPreRIExplorations.mxd 9/17/2018

Legend

- Ⓐ Ambient Air Sampling Location
- Soil Boring
- Ⓐ Indoor Air Sampling Location
- ● Monitoring Well
- ⊕ Soil/Soil Gas Sampling Location
- Soil Gas Sampling Location
- ▚ Test Pit
- 🟧 Building C-23 and C-23 Annex Investigation Area

0 50 100

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes

1. AST = aboveground storage tank
   UST = underground storage tank.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

LANDAU ASSOCIATES

TECT Aerospace Everett, Washington	**Building C-23 and C-23 Annex Pre-RI Exploration Locations**	Figure 7a

G:\Projects\222\057\010\013\RI Work Plan\F07bBuildingC23AOCPreRISoilExceedances.mxd 9/17/2018

Legend

Sampling Location

Building C-23 and C-23 Annex Investigation Area

Not Analyzed

🟨 Analyzed, Detected, No Exceedance

🟩 Analyzed, Not Detected

🟥 Exceedance

0 50 100

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Note
1. TPH = total petroleum hydrocarbons
VOCs = volatile organic compounds.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

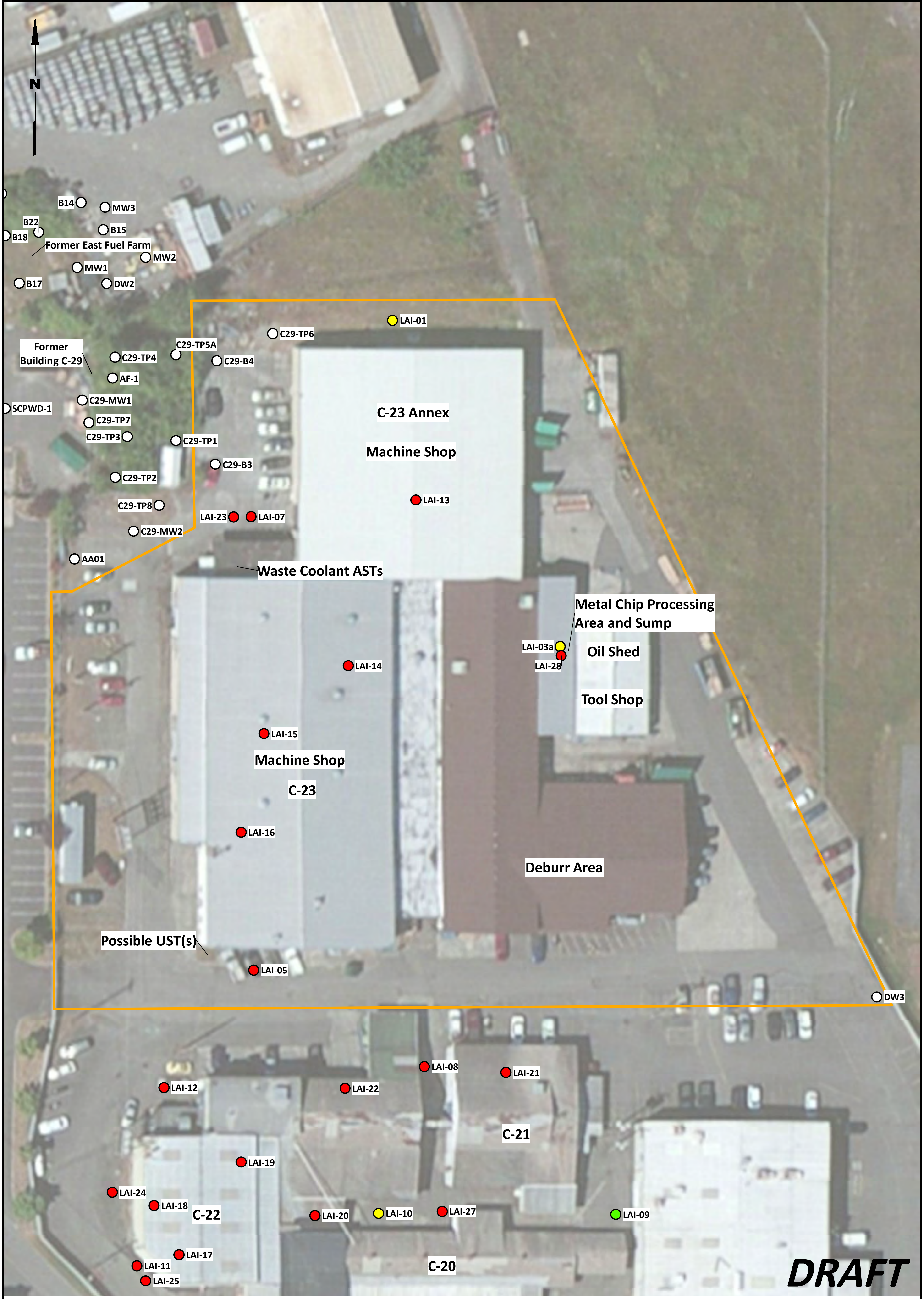
Landau Associates

TECT Aerospace
Everett, Washington

Building C-23 and C-23 Annex
Pre-RI Soil Results

Figure
7b

G:\Projects\222\057\010\013\RI Work Plan\F07cBuildingC23AOCPreRISoilGasExceedances.mxd 9/17/2018



Legend

○ Soil Gas Not Analyzed

🟢 Soil Gas Analyzed, Not Detected

🟡 Soil Gas Analyzed, Detected, No Exceedance

🔴 Soil Gas Exceedance

🟧 Building C-23 and C-23 Annex Investigation Area

0 50 100
Scale in Feet

Note
1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Building C-23 and C-23 Annex
Pre-RI Soil Gas Results

Figure
7c

Notes

1. Detected concentrations of trichloroethene in indoor air exceed the MTCA Method B screening level, but are below the MTCA Method C screening level.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Legend

○ Not Analyzed
🟢 Analyzed, Not Detected
🟡 Analyzed, Detected, No Exceedance
🔴 Exceedance
▭ Building C-23 and C-23 Annex Investigation Area

0 50 100

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Landau Associates

TECT Aerospace
Everett, Washington

Building C-23 and C-23 Annex
Pre-RI Air Results

Figure
7d

G:\Projects\222\057\010\013\RI Work Plan\F07eBuildingC23AOCProposedRIExplorations.mxd 9/17/2018

Legend

- Proposed Soil Sampling Location
- Proposed Soil and Groundwater Sampling Location
- Ambient Air Sampling Location
- Soil Boring
- Indoor Air Sampling Location
- ● Monitoring Well
- ⊕ Soil/Soil Gas Sampling Location
- Soil Gas Sampling Location
- ▨ Test Pit
- Building C-23 and C-23 Annex Investigation Area

0 50 100
Scale in Feet

Note
1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

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Building C-23 and C-23 Annex
Proposed RI Exploration Locations

Figure
7e

Legend

Ⓐ Ambient Air Sampling Location
◕ Soil Boring
Ⓐ Indoor Air Sampling Location
● Monitoring Well
⊕ Soil/Soil Gas Sampling Location
▚ Test Pit
▭ Former Building C-29/Former East Fuel Farm Investigation Area

0 30 60

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes
1. UST = underground storage tank.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

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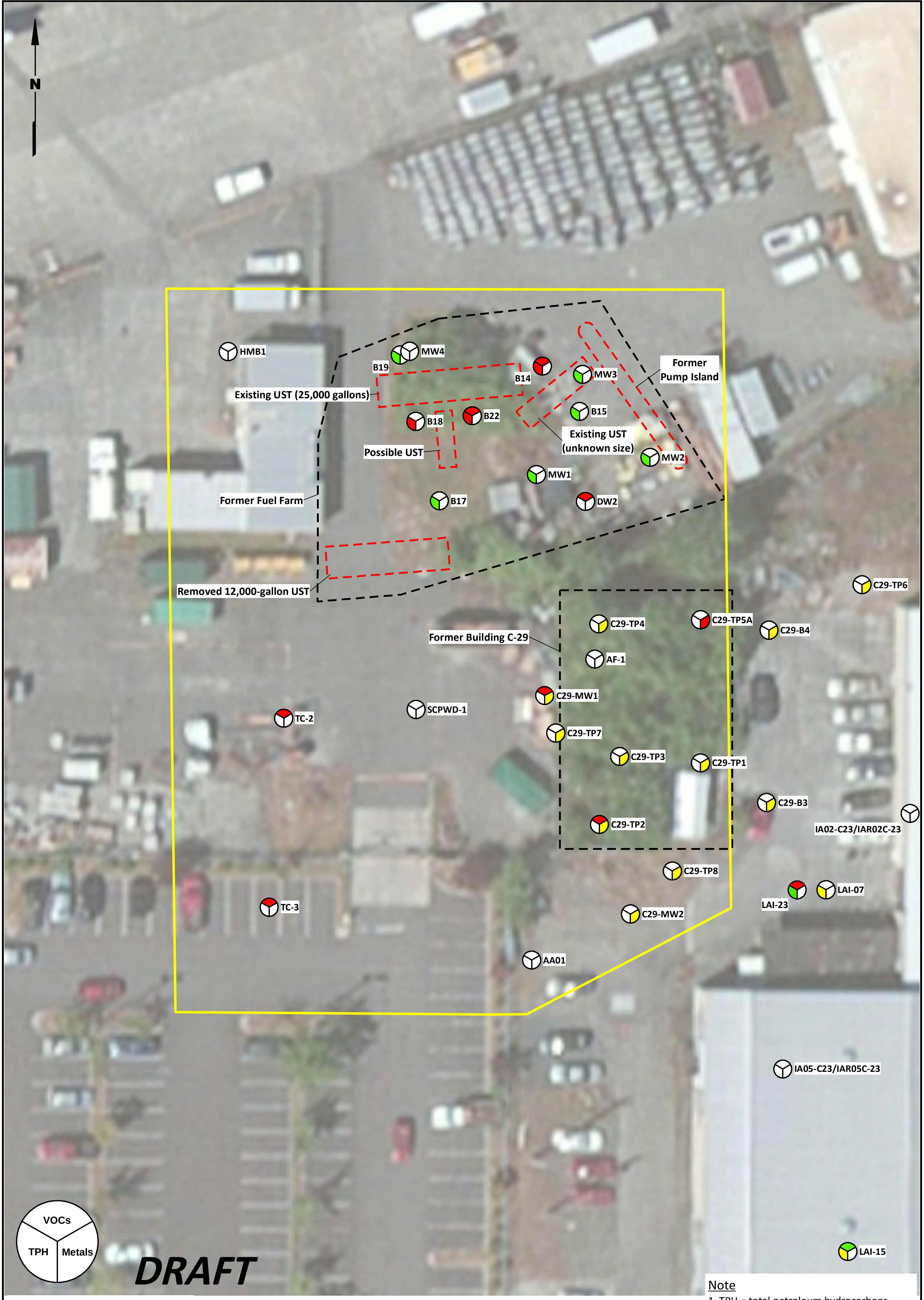
Everett, Washington

Former Building C-29/Former Fuel Farm Pre-RI Exploration Locations

Figure 8a

G:\Projects\222\057\010\013\RI Work Plan\F08bFormerBuildingC-29AOCPreRISoilExceedances.mxd 9/17/2018



Legend

Sampling Location

Not Analyzed

Analyzed, Detected, No Exceedance

Analyzed, Not Detected

🟥 Exceedance

🟨 Former Building C-29/Former East Fuel Farm Investigation Area

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Note
1. TPH = total petroleum hydrocarbons
UST = underground storage tank
VOCs = volatile organic compounds.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 30 60
Scale in Feet

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Former Building C-29/Former Fuel Farm
Pre-RI Soil Results

Figure
8b

G:\Projects\222\057\010\013\RI Work Plan\F08cFormerBuildingC-29AOCPreRIGWExceedances.mxd 9/17/2018

Legend

○ Sampling Location

◻ Not Analyzed

🟨 Analyzed, Detected, No Exceedance

🟩 Analyzed, Not Detected

🟥 Exceedance

🟨 Former Building C-29/Former East Fuel Farm Investigation Area

0 30 60

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes

1. TPH = total petroleum hydrocarbons
   UST = underground storage tank
   VOCs = volatile organic compounds.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Former Building C-29/Former Fuel Farm
Pre-RI Groundwater Results

Figure
8c

G:\Projects\222\057\010\013\RI Work Plan\F08dFormerBuildingC-29AOCProposedRIExplorations.mxd 9/17/2018

Legend

●	Proposed Soil Sampling Location
●	Proposed Soil and Groundwater Sampling Location
●	Soil, Soil Gas, and Groundwater Sampling Location
Ⓐ	Ambient Air Sampling Location
⊕	Soil Boring
Ⓐ	Indoor Air Sampling Location
●	Monitoring Well
⊕	Soil/Soil Gas Sampling Location
▨	Test Pit
☐	Former Building C-29/Former East Fuel Farm Investigation Area

0 30 60
Scale in Feet

Notes
1. UST = underground storage tank
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

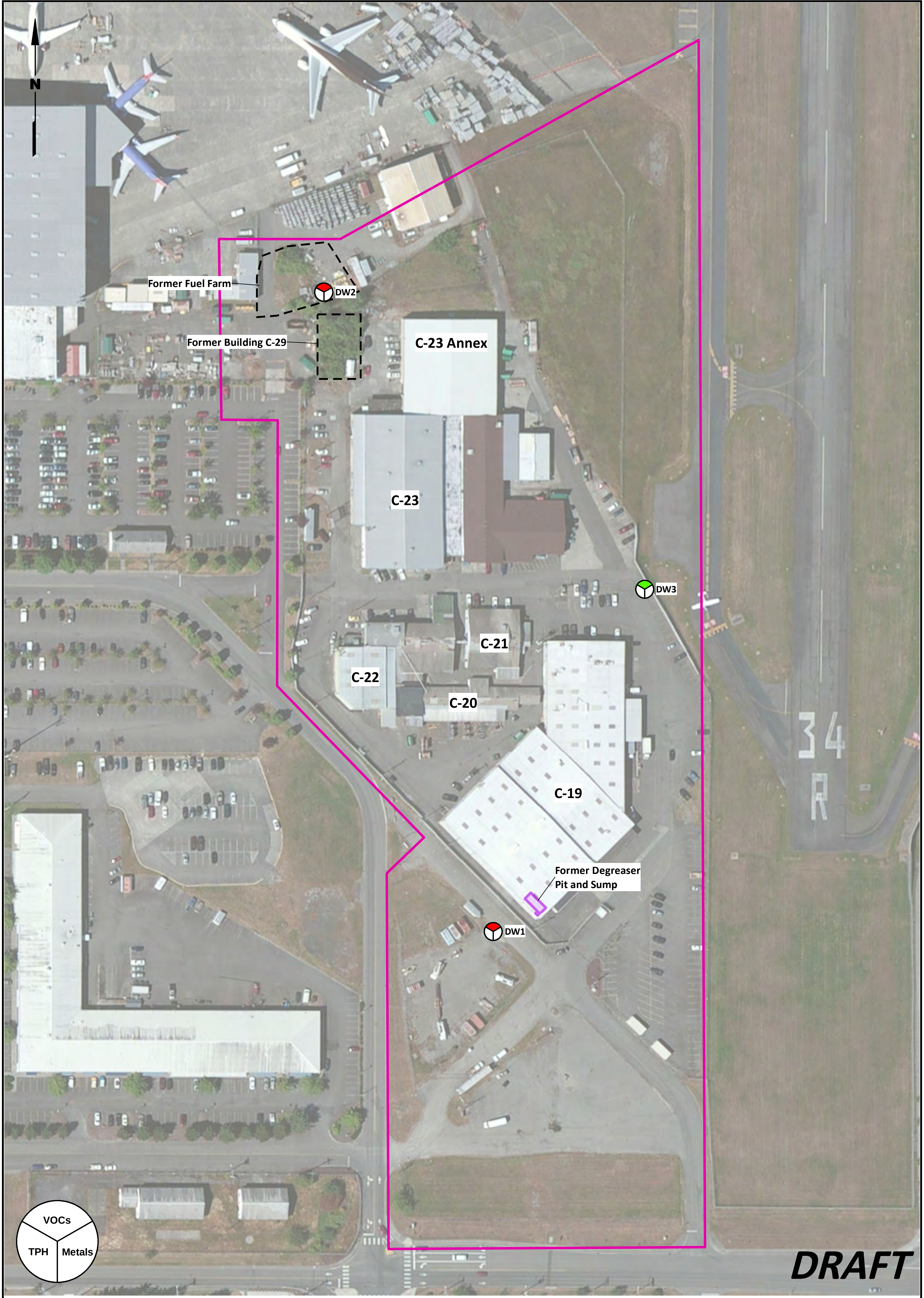
LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Former Building C-29/Former Fuel Farm
Proposed RI Exploration Locations

Figure
8d

G:\Projects\222\057\010\013\RI Work Plan\F09aDeepAquiferAOCPreRISoilExceedances.mxd 9/17/2018



Legend

Sampling Location	Not Analyzed	🟩 Analyzed, Not Detected
🟪 Deep Aquifer Investigation Area	🟨 Analyzed, Detected, No Exceedance	🟥 Exceedance

Notes

1. TPH = total petroleum hydrocarbons
   VOCs = volatile organic compounds.
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

0 120 240

Scale in Feet

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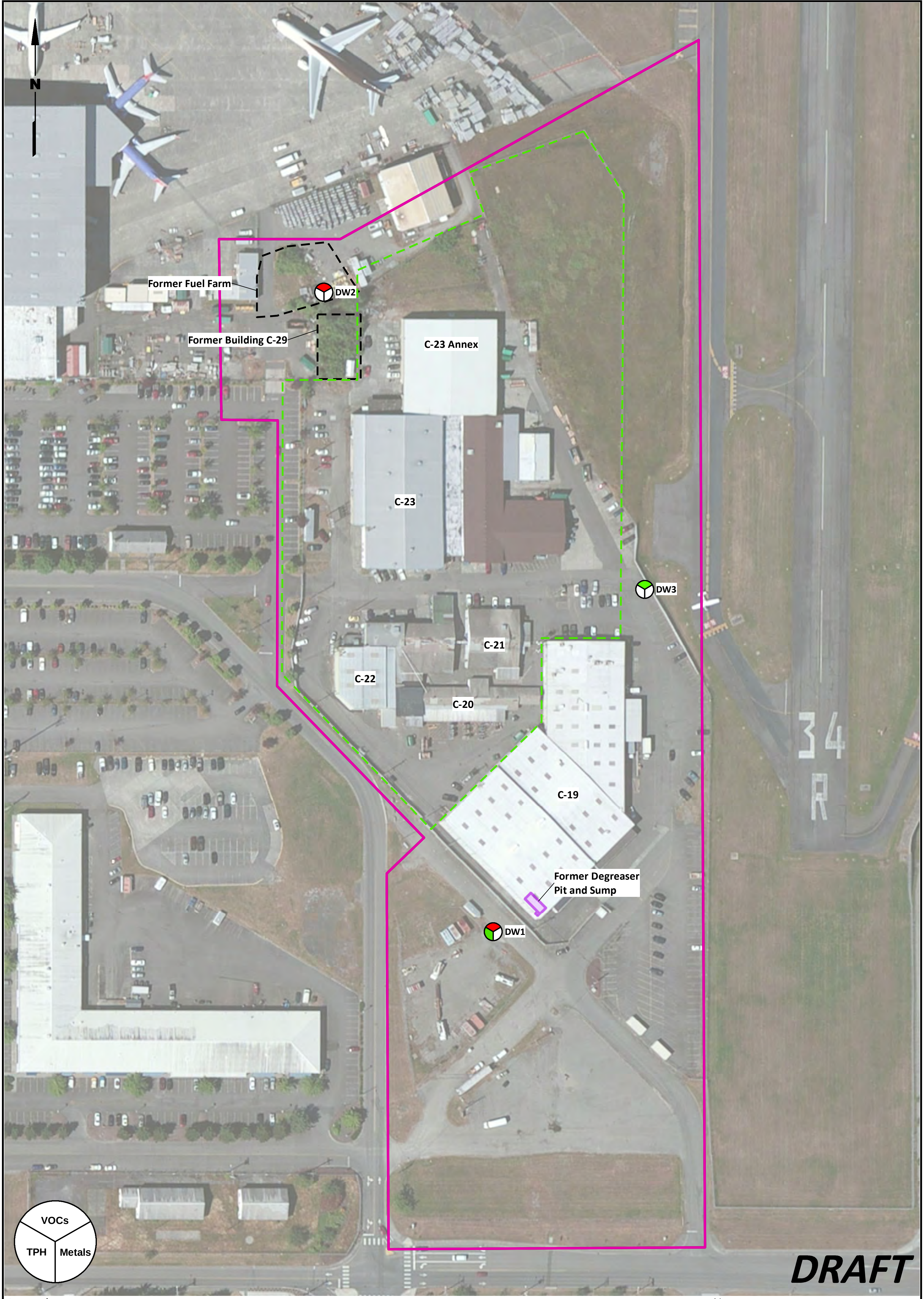
Everett, Washington

Deep Aquifer
Pre-RI Soil Results

Figure
9a

G:\Projects\222\057\010\013\RI Work Plan\F09bDeepAquiferAOCPreRIGWExceedances.mxd 9/17/2018



Legend

Sampling Location

▭ Deep Aquifer Investigation Area

☐ Not Analyzed

🟨 Analyzed, Detected, No Exceedance

🟩 Analyzed, Not Detected

🟥 Exceedance

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Notes
1. TPH = total petroleum hydrocarbons
VOCs = volatile organic compounds
2. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 120 240
Scale in Feet

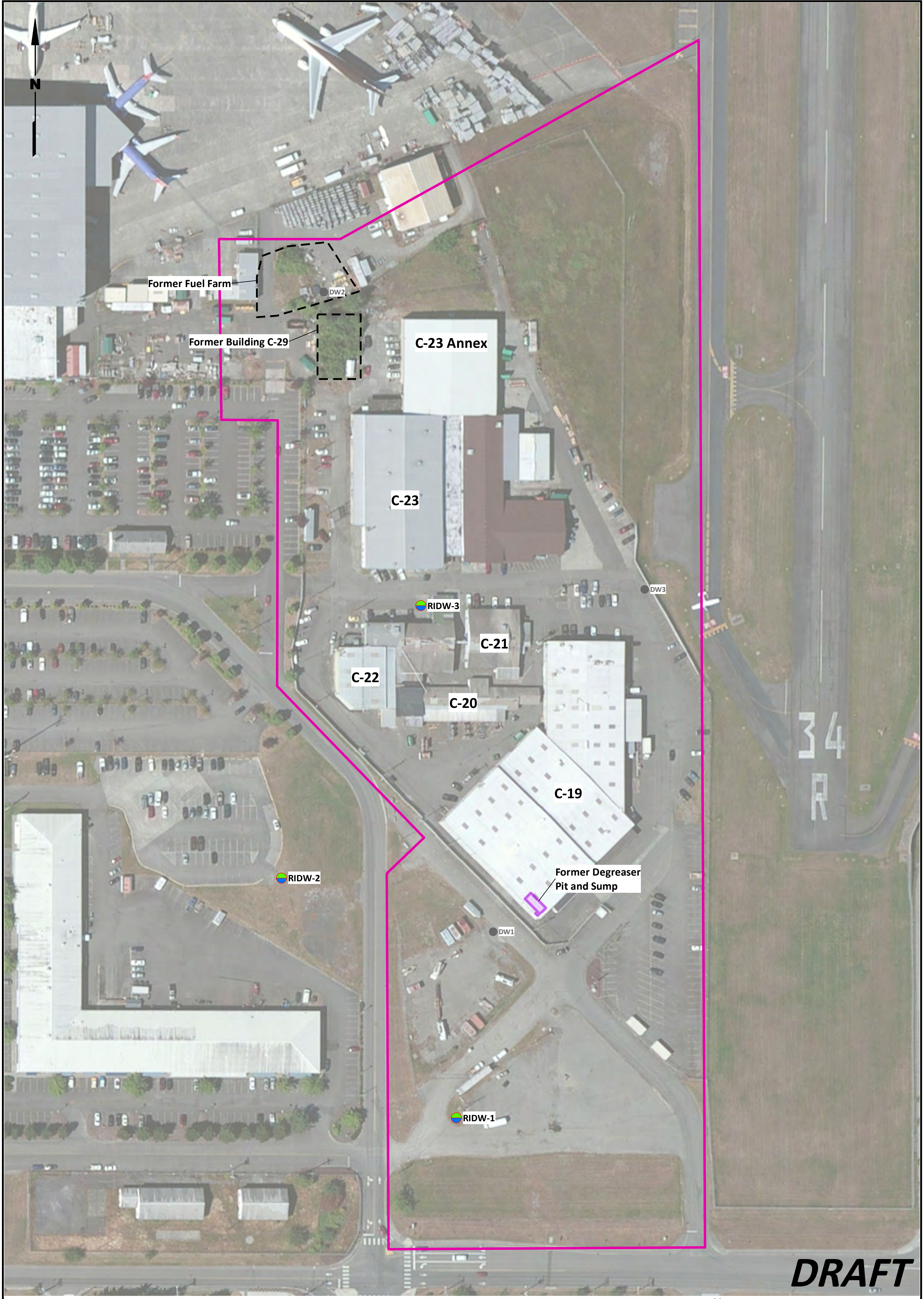
LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Deep Aquifer
Pre-RI Groundwater Results

Figure
9b

G:\Projects\222\057\010\013\RI Work Plan\F09cDeepAquiferAOCProposedRIExplorations.mxd 9/17/2018



Legend

- ● Proposed Soil and Groundwater Sampling Location
- ● Monitoring Well
- ▭ Deep Aquifer Investigation Area

0 120 240

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

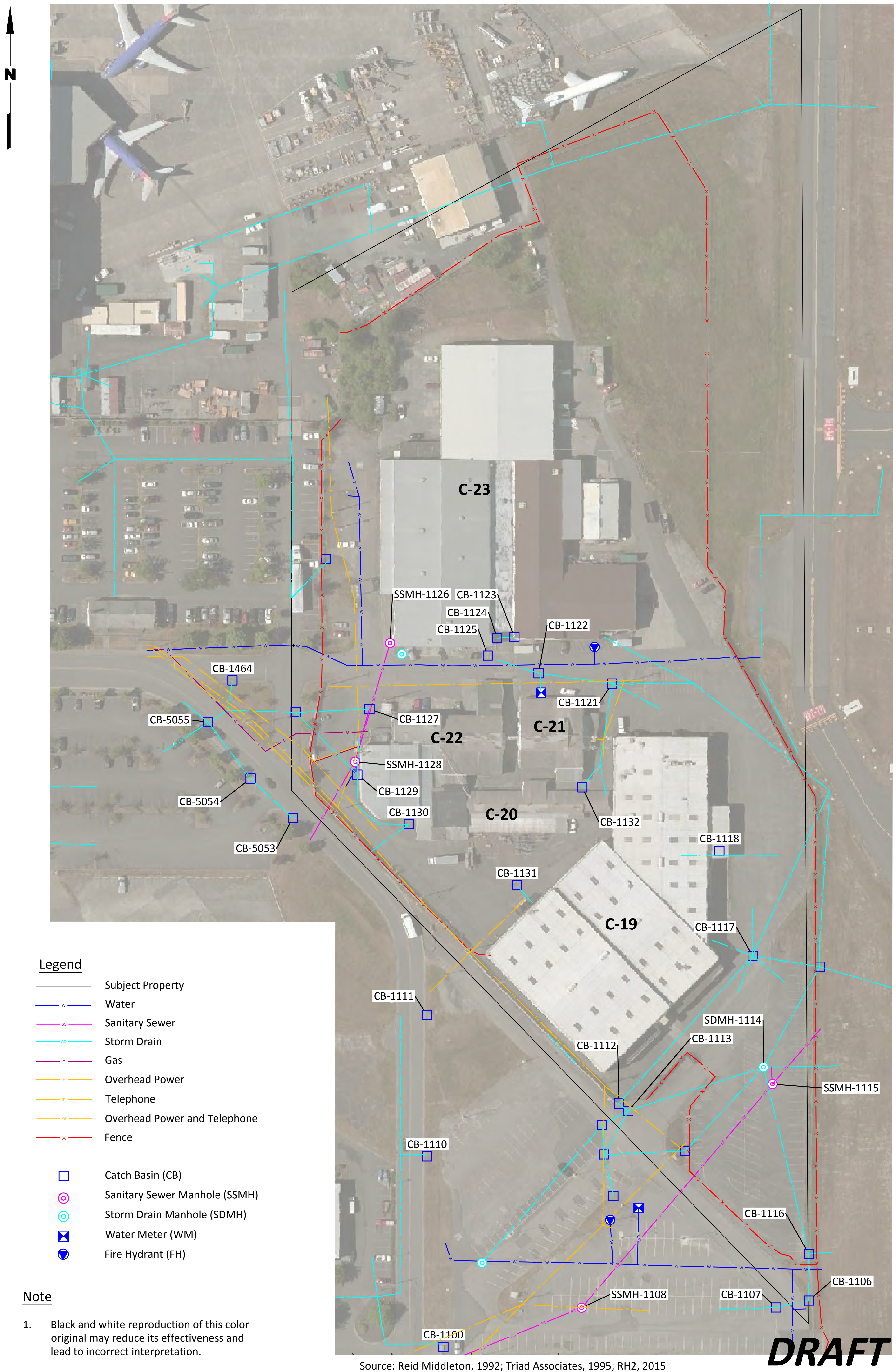
Landau Associates

TECT Aerospace
Everett, Washington

Deep Aquifer
Proposed RI Exploration Locations

Figure
9c

Landau Associates | G:\Projects\222\057\010\013\RI Work Plan\F10Utilities.dwg | 9/17/2018 9:42 PM



Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

Source: Reid Middleton, 1992; Triad Associates, 1995; RH2, 2015

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Utilities Map

Figure
10

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Table 1
C-19 Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	VOCs											
			Analyte	1,1,1-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene	Acetone	Carbon Disulfide	cis-1,2-Dichloroethene	Methyl Ethyl Ketone	Methylene Chloride	Tetrachloroethene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	84.3	2.61	2.46	2,070	266	5.15	48,000,000	1.48	2.76	32.5	0.206	0.009
Location	Sample Depth	Depth Units	Sample Date												
C19-TP1	0	FT	2/14/1994	**44**	**2.3**	**2.2**	**6.6**	1.1 U	**8.1**	ND	5.7 U	1.1 U	ND	**220**	ND
C19-TP1	0.9	FT	2/14/1994	**39**	**2**	ND	ND	1.1 U	**11**	ND	5.7 U	1.1 U	ND	**590**	ND
C19-TP2	0.1	FT	2/14/1994	**5.4**	ND	ND	ND	1.1 U	ND	ND	5.2 U	1.1 U	ND	**19**	ND
C19-TP2	0.9	FT	2/14/1994	ND	ND	ND	ND	1.1 U	ND	ND	5.6 U	1.1 U	ND	**27**	ND
DW1	57.5	FT	12/12/2000	--	--	--	--	--	10 U	--	--	--	10 U	**10**	--
DW1	77	FT	12/12/2000	--	--	--	--	--	10 U	--	--	--	10 U	10 U	--
DW1	97.5	FT	12/12/2000	--	--	--	--	--	10 U	--	--	--	10 U	10 U	--
DW1	117	FT	12/12/2000	--	--	--	--	--	10 U	--	--	--	10 U	10 U	--
DW1	137	FT	12/12/2000	--	--	--	--	--	10 U	--	--	--	10 U	10 U	--
GP13	9	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP15	3	FT	3/20/2003	--	--	--	--	--	--	--	**22 B**	--	--	10 U	--
GP17	8	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	**43**	--
GP18	2	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP18	9	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	**59**	--
GP3	4.5	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP3	8	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	**63**	--
GP4	3	FT	3/20/2003	--	--	--	--	--	--	--	**26 B**	--	--	10 U	--
GP5	3	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP5	8	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP6	2	FT	3/20/2003	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP7	2	FT	3/21/2013	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP8	2	FT	3/21/2013	--	--	--	--	--	--	--	20 U	--	--	10 U	--
GP9	2	FT	3/21/2013	--	--	--	--	--	--	--	20 U	--	--	10 U	--

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Table 1
C-19 Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	VOCs											
			Analyte	1,1,1-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene	Acetone	Carbon Disulfide	cis-1,2-Dichloroethene	Methyl Ethyl Ketone	Methylene Chloride	Tetrachloroethene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	84.3	2.61	2.46	2,070	266	5.15	48,000,000	1.48	2.76	32.5	0.206	0.009
SCPWD-2	8.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**64.9**	--
SCPWD-2	11.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**69.4**	--
SCPWD-2	16.5	FT	12/23/1996	--	--	--	--	--	**62.5**	--	--	--	--	**1,050**	--
SCPWD-3	8.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**1,720**	--
SCPWD-3	11.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**1,350**	--
SCPWD-3	13.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**463**	--
SCPWD-3	16.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**99.8**	--
SCPWD-3	18.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	50 U	--
SCPWD-3	21.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**1,990**	--
SCPWD-3	23.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**2,100**	--
SCPWD-3	28.5	FT	12/23/1996	--	--	--	--	--	50 U	--	--	--	--	**4,300**	--
SCPWD-3	33.5	FT	12/23/1996	--	--	--	--	--	**50**	--	--	--	--	**1,250**	--
SCPWD-4	8.5	FT	12/13/1996	--	--	--	--	--	50 U	--	--	--	--	50 U	--
SCPWD-4	13.5	FT	12/13/1996	--	--	--	--	--	50 U	--	--	--	--	50 U	--
SCPWD-4	18.5	FT	12/13/1996	--	--	--	--	--	50 U	--	--	--	--	50 U	--
SU2-FL-0.8	0.8	FT	7/27/1995	ND	ND	ND	**110**	ND	**44**	ND	**41**	ND	ND	**4,700**	ND
SU2-NE-0.8	0.8	FT	7/27/1995	ND	ND	ND	**59**	ND	**57**	ND	**86**	ND	ND	**4,400**	ND
SU2-NW-0.8	0.8	FT	7/27/1995	ND	ND	ND	**78**	ND	**19**	ND	**78**	ND	ND	**1,900**	ND
SU-FL-0.5	0.5	FT	7/27/1995	ND	**47**	**6.9**	ND	ND	**47**	**14**	ND	ND	**2.6**	**2,300**	ND
SU-FL-1.0	1.0	FT	7/27/1995	**29**	**73**	ND	**220**	ND	**100**	ND	**28**	ND	ND	**3,200**	ND
SU-NE-0.4	0.4	FT	7/27/1995	ND	**13**	**1.2**	ND	**1.6**	**31**	ND	ND	**2.2**	**2.1**	**8,900**	ND
SU-NE-1.0	1.0	FT	7/27/1995	ND	**24**	ND	**56**	ND	**53**	ND	ND	ND	ND	**10,000**	ND
SU-NW-0.5	0.5	FT	7/27/1995	ND	ND	ND	ND	ND	**54**	ND	ND	ND	ND	**4,400**	ND
SU-NW-1.0	1.0	FT	7/27/1995	ND	ND	ND	**87**	ND	**47**	ND	ND	ND	ND	**4,100**	ND
SU-SE-0.5	0.5	FT	7/27/1995	ND	**1.2**	ND	ND	ND	**46**	ND	ND	ND	ND	**1,700**	ND
SU-SE-1.1	1.1	FT	7/27/1995	ND	ND	ND	ND	ND	**6.7**	ND	ND	ND	ND	**69**	ND
SU-SW-0.5	0.5	FT	7/27/1995	**1.1**	ND	ND	ND	ND	**8.7**	ND	ND	ND	ND	**160**	ND
SU-SW-1.0	1.0	FT	7/27/1995	ND	**17**	**4.8**	ND	ND	**53**	**7.4**	ND	ND	**1.9**	**5,700**	**4.7**

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

B = Analyte was detected in the associated method blank.

Bold = detected compound

-- = not analyzed

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

FT = feet

µg/kg = micrograms per kilogram

ND = not detected

VOC = volatile organic compound

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Table 2
C-19 Historical Data – Detected Constituents in Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	Metals		VOCs											
	Analyte	Chromium, Total	Chromium, Dissolved	1,1,1-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene	4-Methyl-2-pentanone	Acetone	cis-1,2-Dichloroethene	Methyl Ethyl Ketone	Tetrachloro-ethene	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride
	Unit of Measurement	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
	Screening Level	100	100	200	7.68	7	640	7200	16	4,800	5	640	100	0.54	0.2
Location	Sample Date														
C19-TP1	2/14/1994	--	--	**230**	**32**	**21**	**5.8**	**17**	**94**	**160**	1 U	**1.6**	**39**	**15,000**	**5.1 M**
DW1	12/28/1999	--	--	5 U	--	--	--	--	5 U	--	--	--	--	**8**	--
DW1	3/8/2000	--	--	5 U	--	--	--	--	**5**	--	--	--	--	**62**	--
DW1	10/24/2001	--	--	--	--	--	--	--	--	--	--	1 U	--	--	--
DW1	10/17/2003	--	--	5 U	--	--	--	--	5 U	--	--	--	--	**81**	--
GP1	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP2	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP3	3/20/2003	--	--	2 U	2 U	2 U	--	--	**100**	--	--	--	**20**	**240**	2 U
GP4	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP5	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP6	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP7	3/21/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP8	3/21/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP9	3/21/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP10	3/21/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP11	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP12	3/20/2003	--	--	**7**	**6**	**3**	--	--	**17**	--	--	--	2 U	**100**	2 U
GP14	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP15	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP16	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
GP17	3/20/2003	--	--	2 U	2 U	2 U	--	--	**130**	--	--	--	**3**	**1,300**	2 U
GP18	3/20/2003	--	--	2 U	2 U	**4**	--	--	**220**	--	--	--	**10**	**5,000**	**44**
GP19	3/20/2003	--	--	2 U	2 U	2 U	--	--	**3**	--	--	--	2 U	**24**	2 U
GP20	3/20/2003	--	--	2 U	2 U	2 U	--	--	2 U	--	--	--	2 U	2 U	2 U
SCPWD-2	3/9/1999	**40**	10 U	--	--	**24**	--	--	**2,500**	--	4 U	--	**18**	**39,000**	**9**
SCPWD-2	10/17/2003	--	--	--	--	5 U	--	--	**540**	--	4 U	--	**4**	**4,300**	5 U
SCPWD-3	3/9/1999	10 U	10 U	--	--	**110**	--	--	**4,200**	--	**12**	--	**18**	**140,000**	**68**
SCPWD-3	10/17/2003	--	--	--	--	**74**	--	--	**3,700**	--	4 U	--	**28**	**100,000**	**61**
SCPWD-4	3/9/1999	10 U	10 U	--	--	5 U	--	--	**260**	--	4 U	--	**160**	**580**	**82**
SCPWD-4	3/21/2003	--	--	--	--	5 U	--	--	**15**	--	4 U	--	**6**	**8**	5 U
SCPWD-4	10/17/2003	--	--	--	--	5 U	--	--	**200**	--	4 U	--	**37**	**190**	5 U
SU2-NE-W	8/7/1995	--	--	ND	ND	ND	ND	ND	**670**	ND	ND	ND	ND	**53,000**	ND
SU-FL-W	8/7/1995	--	--	**130**	**420**	**70**	ND	ND	**340**	ND	ND	ND	ND	**98,000**	ND
SW-1.0-W	8/7/1995	--	--	**160**	**320**	**88**	ND	ND	**360**	ND	ND	ND	ND	**39,000**	ND

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Table 2
C-19 Historical Data – Detected Constituents in Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Notes:

M = laboratory flag indicating low spectral match
U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
Bold = detected compound
-- = not analyzed

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

µg/L = micrograms per liter
ND = not detected
VOC = volatile organic compound

Table 3
C-20, C-21, C1-22 Historical Data - Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	**TPH**		**VOCs**	
			Analyte	Petroleum Hydrocarbons C12-C24 (DRO)	Petroleum Hydrocarbons C24-C40 (ORO)	cis-1,2-Dichloroethene	Trichloroethene
			Unit of Measurement	mg/kg	mg/kg	µg/kg	µg/kg
			Screening Level	2,000	2,000	5.15	0.206
Location	Sample Depth	Depth Units	Sample Date				
LAI-10	1	FT	5/2/2017	120 U	**4,200**	--	--
LAI-12	3	FT	5/2/2017	25 U	50 U	--	--
LAI-17	1.7	FT	10/5/2017	25 U	50 U	10 U	10 U
LAI-18	1.8	FT	10/5/2017	25 U	50 U	10 U	10 U
LAI-19	2.4	FT	10/5/2017	**47**	50 U	10 U	10 U
LAI-20	1.2	FT	10/5/2017	25 U	50 U	10 U	10 U
LAI-21	1.5	FT	10/5/2017	25 U	**52**	10 U	10 U
LAI-22	1.5	FT	10/5/2017	25 U	50 U	10 U	10 U
LAI-24	10.75	FT	10/9/2017	25 U	**320**	10 U	10 U
LAI-25	15	FT	10/5/2017	25 U	50 U	**320**	**4,000**
LAI-26	6.5	FT	10/5/2017	25 U	**80**	10 U	**40**
LAI-26	9.5	FT	10/5/2017	--	--	**21**	**1,400**
LAI-27	8	FT	10/6/2017	25 U	50 U	**19**	**3,800**

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

-- = not analyzed

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

DRO - diesel-range organics	mg/kg = milligrams per kilogram	ORO = oil-range organics
FT = feet	VOC = volatile organic compound	TPH = total petroleum hydrocarbons

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Table 4
C-20, C-21, C1-22 Historical Data – Detected Constituents in Soil Gas
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs														
	Analyte	1,1-Dichloroethene	1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	1,3-Butadiene	2,2,4-Trimethylpentane	4-Ethyltoluene	4-Methyl-2-pentanone	Acetone	Benzene	Carbon Disulfide	Chloroform	cis-1,2-Dichloroethene	Cyclohexane	Ethanol	Ethylbenzene
	Unit of Measurement	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$	$\mu g/m^3$
	Screening Level (a)	3,050	107	--	2.78	--	--	45,700	--	10.7	10,700	3.62	--	--	--	15,200
Location	Sample Date															
LAI-08	5/2/2017	4.4 U	5.4 U	5.4 U	2.4 U	**11**	5.4 U	4.5 U	**59**	**15**	**17**	5.4 U	4.4 U	**200**	**27**	**5.4**
LAI-09	5/3/2017	4.1 U	5.0 U	5.0 U	2.3 U	4.8 U	5.0 U	4.2 U	24 U	3.3 U	13 U	5.0 U	4.1 U	3.5 U	7.7 U	4.4 U
LAI-10	5/2/2017	4.1 U	5.1 U	5.1 U	2.3 U	4.8 U	5.1 U	4.2 U	**33**	3.3 U	13 U	5.0 U	4.1 U	3.5 U	**34**	4.5 U
LAI-11	5/2/2017	39 U	49 U	49 U	22 U	46 U	49 U	41 U	240 U	32 U	**160**	48 U	**440**	34 U	75 U	43 U
LAI-12	5/2/2017	4.2 U	5.2 U	5.2 U	**120**	**6.2**	5.2 U	4.3 U	**66**	**16**	13 U	5.1 U	4.2 U	**22**	**33**	4.6 U
LAI-17	10/4/2017	12 U	15 U	15 U	6.7 U	14 U	15 U	12 U	36 U	9.7 U	47 U	15 U	**300**	10 U	**710**	13 U
LAI-18	10/4/2017	12 U	15 U	15 U	6.7 U	14 U	15 U	12 U	36 U	9.7 U	47 U	15 U	**1,600**	10 U	**1,800**	13 U
LAI-19	10/4/2017	1.8 U	**30**	**8.2**	1.0 U	2.2 U	**21**	**9.2**	**170**	**5.9**	7.2 U	**25**	1.8 U	1.6 U	**430**	**6.6**
LAI-20	10/4/2017	1.9 U	**30**	**8.4**	1.0 U	**15**	**25**	**9.0**	**3,500 E**	**7.7**	7.4 U	**17**	**120**	**5.1**	**300**	**7.2**
LAI-21	10/4/2017	3.6 U	**52**	**17**	2.0 U	4.3 U	**37**	3.7 U	**850**	**9.6**	14 U	4.5 U	3.6 U	**12**	**290**	4.0 U
LAI-22	10/4/2017	3.5 U	**72**	**19**	2.0 U	4.1 U	**38**	3.6 U	**2,300 E**	2.8 U	14 U	**11**	3.5 U	3.0 U	**790**	3.8 U
LAI-24	10/6/2017	4.2 U	5.2 U	5.2 U	2.3 U	4.9 U	5.2 U	**17**	12 U	3.3 U	16 U	**64**	4.2 U	**15**	**290**	4.6 U
LAI-25	10/6/2017	**130**	30 U	30 U	**71**	29 U	30 U	25 U	74 U	20 U	97 U	30 U	**1,500**	21 U	**1,300**	27 U
LAI-26	10/6/2017	**480**	47 U	47 U	**51**	**57**	47 U	39 U	85 U	30 U	110 U	46 U	**13,000**	**52**	**480 J**	41 U
LAI-27	10/6/2017	1.8 U	**10**	2.2 U	**28**	**9.8**	**11**	**11**	**94**	**17**	6.9 U	2.2 U	**170**	**59**	**630**	**16**

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Table 4
C-20, C-21, C1-22 Historical Data – Detected Constituents in Soil Gas
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs											
	Analyte	Isopropanol	m-&p-Xylenes	Methyl Ethyl Ketone	n-Heptane	n-Hexane	o-Xylene	Tetrachloroethene	Tetrahydrofuran	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride
	Unit of Measurement	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3	μg/m^3
	Screening Level (a)	--	1,520	76,200	--	10,700	1,520	321	--	76,200	--	12.3	9.33
Location	Sample Date												
LAI-08	5/2/2017	11 U	**20**	13 U	**140**	**350**	**6.7**	7.5 U	3.2 U	**55**	4.4 U	5.9 U	2.8 U
LAI-09	5/3/2017	10 U	4.4 U	12 U	4.2 U	3.6 U	4.4 U	7.0 U	3.0 U	3.9 U	4.1 U	5.5 U	2.6 U
LAI-10	5/2/2017	10 U	4.5 U	12 U	4.2 U	3.6 U	4.5 U	7.0 U	3.0 U	**9.1**	4.1 U	5.5 U	2.6 U
LAI-11	5/2/2017	98 U	43 U	120 U	**42**	**60**	43 U	67 U	29 U	**150**	39 U	53 U	**5,400**
LAI-12	5/2/2017	10 U	**5.3**	**17**	**21**	**85**	4.6 U	7.1 U	3.1 U	**24**	4.2 U	5.6 U	2.7 U
LAI-17	10/4/2017	37 U	13 U	45 U	12 U	11 U	13 U	21 U	9.0 U	11 U	12 U	**12,000**	7.8 U
LAI-18	10/4/2017	**120**	13 U	45 U	12 U	11 U	13 U	21 U	9.0 U	11 U	**51**	**16,000**	7.8 U
LAI-19	10/4/2017	**33**	**25**	**120**	1.9 U	**11**	**9.6**	3.2 U	**13**	**25**	1.8 U	**29**	1.2 U
LAI-20	10/4/2017	**32**	**29**	**62**	2.0 U	**23**	**11**	**8.2**	**7.6**	**23**	**5.8**	**1,300**	1.2 U
LAI-21	10/4/2017	**26**	**17**	**52**	**37**	**38**	4.0 U	6.2 U	**8.1**	**19**	3.6 U	**410**	2.3 U
LAI-22	10/4/2017	**63**	**12**	**36**	**13**	**21**	3.8 U	**42**	2.6 U	**13**	3.5 U	**29**	2.2 U
LAI-24	10/6/2017	13 U	**15**	15 U	4.3 U	**12**	4.6 U	7.1 U	3.1 U	**26**	4.2 U	5.6 U	2.7 U
LAI-25	10/6/2017	76 U	27 U	92 U	25 U	22 U	27 U	42 U	18 U	23 U	**170**	**29,000**	16 U
LAI-26	10/6/2017	88 U	41 U	100 U	39 U	**110**	41 U	**130**	28 U	36 U	**140**	**74,000**	**31**
LAI-27	10/6/2017	**92**	**58**	**29**	**60**	**73**	**16**	3.0 U	1.3 U	**170**	1.8 U	**34**	**59**

Notes:

(a) MTCA Method B Screening Level

E = Exceeds the calibration curve of the instrument. Reported concentration is estimated.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

Bold = detected compound

🟩 **Green Box** = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

μg/m^3 = micrograms per cubic meter

MTCA = Model Toxics Control Act

VOC = volatile organic compound

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Table 5
C-23 Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	TPH		VOCs
			Analyte	Petroleum Hydrocarbons C12-C24 (DRO)	Petroleum Hydrocarbons C24-C40 (ORO)	cis-1,2-Dichloroethene
			Unit of Measurement	mg/kg	mg/kg	µg/kg
			Screening Level	2,000	2,000	5.15
Location	Sample Depth	Depth Units	Sample Date			
DW3	7	FT	12/12/2000	--	--	10 U
DW3	36	FT	12/12/2000	--	--	10 U
DW3	66	FT	12/12/2000	--	--	10 U
DW3	76	FT	12/12/2000	--	--	10 U
DW3	136	FT	12/12/2000	--	--	10 U
DW3	151	FT	12/12/2000	--	--	10 U
LAI-03a	3	FT	5/2/2017	25 U	50 U	--
LAI-05	9	FT	5/2/2017	25 U	50 U	--
LAI-07	1	FT	5/3/2017	25 U	**76**	--
LAI-13	1.2	FT	10/5/2017	25 U	**460**	10 U
LAI-14	1.2	FT	10/5/2017	25 U	50 U	10 U
LAI-15	1.7	FT	10/5/2017	**27**	**70**	10 U
LAI-16	2.1	FT	10/5/2017	250 U	🟩 **6,900**	10 U
LAI-23	16.5	FT	10/5/2017	25 U	50 U	🟩 **36**
LAI-28	17.75	FT	10/9/2017	25 U	50 U	10 U

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

-- = not analyzed

🟩 **Green Box** = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

DRO = diesel-range organics

FT = feet

mg/kg = milligrams per kilogram

ORO = oil-range organics

TPH = total petroleum hydrocarbons

VOC = volatile organic compound

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Table 6
C-23 Historical Data – Detected Constituents in Soil Gas
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs													
	Analyte	1,1-Dichloroethane	1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	1,3-Butadiene	2,2,4-Trimethylpentane	2-Hexanone	4-Ethyltoluene	4-Methyl-2-pentanone	Acetone	Benzene	Carbon Disulfide	Carbon Tetrachloride	Chloroform	cis-1,2-Dichloroethene
	Unit of Measurement	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3
	Screening Level (a)	52.1	107	--	2.78	--	--	--	45,700	--	10.7	10,700	13.9	3.62	--
Location	Sample Date														
LAI-01	5/3/2017	4.1 U	5.0 U	5.0 U	2.2 U	4.8 U	17 U	5.0 U	4.2 U	**27**	3.2 U	13 U	6.4 U	5.0 U	4.0 U
LAI-03a	5/2/2017	4.1 U	5.0 U	5.0 U	2.2 U	4.8 U	17 U	5.0 U	4.2 U	**26**	3.2 U	13 U	6.4 U	5.0 U	4.0 U
LAI-05	5/2/2017	4.3 U	5.2 U	5.2 U	**150**	**5.1**	17 U	5.2 U	4.3 U	**57**	**30**	**13**	6.7 U	5.2 U	4.2 U
LAI-07	5/3/2017	5.2 U	6.3 U	6.3 U	2.8 U	**7.6**	21 U	6.3 U	5.2 U	**180**	**14**	**17**	8.0 U	6.2 U	**16**
LAI-13	10/4/2017	20 U	24 U	24 U	11 U	23 U	99 U	24 U	20 U	57 U	15 U	75 U	30 U	24 U	**59**
LAI-14	10/4/2017	2.0 U	**33**	**10**	**3.2**	2.3 U	9.9 U	**22**	**28**	**200**	**7.0**	7.5 U	**7.8**	**16**	1.9 U
LAI-15	10/4/2017	1.9 U	**7.0**	2.3 U	1.0 U	2.2 U	9.5 U	**5.8**	**5.5**	**140**	**11**	7.2 U	2.9 U	**23**	1.8 U
LAI-16	10/4/2017	1.8 U	**5.6**	2.2 U	**70**	2.1 U	**70**	**5.7**	**880**	**730**	**73**	**170**	2.8 U	**34**	1.8 U
LAI-23	10/6/2017	**64**	9.3 U	9.3 U	**31**	8.9 U	39 U	9.3 U	7.8 U	**150**	**46**	30 U	12 U	9.3 U	**85**
LAI-28	10/6/2017	1.9 U	2.3 U	2.3 U	**6.1**	**29**	9.7 U	2.3 U	**6.8**	**540**	**12**	**53**	3.0 U	**7.2**	**9.3**

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Table 6
C-23 Historical Data – Detected Constituents in Soil Gas
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs													
	Analyte	Cyclohexane	Ethanol	Ethylbenzene	Isopropanol	m-&p-Xylenes	Methyl Ethyl Ketone	n-Heptane	n-Hexane	o-Xylene	Tetrachloroethene	Tetrahydrofuran	Toluene	Trichloroethene	Vinyl Chloride
	Unit of Measurement	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3	µg/m^3
	Screening Level (a)	--	--	15,200	--	1,520	76,200	--	10,700	1,520	321	--	76,200	12.3	9.33
Location	Sample Date														
LAI-01	5/3/2017	3.5 U	**7.9**	4.4 U	10 U	4.4 U	12 U	4.2 U	3.6 U	4.4 U	6.9 U	3.0 U	3.8 U	5.5 U	2.6 U
LAI-03a	5/2/2017	3.5 U	**26**	4.4 U	10 U	4.4 U	12 U	4.2 U	3.6 U	4.4 U	6.9 U	3.0 U	**8.0**	5.5 U	2.6 U
LAI-05	5/2/2017	**16**	8.0 U	4.6 U	10 U	**7.0**	**13**	**20**	**47**	4.6 U	7.2 U	3.1 U	**30**	5.7 U	2.7 U
LAI-07	5/3/2017	**28**	**25**	5.6 U	12 U	5.6 U	**45**	**28**	**57**	5.6 U	8.7 U	3.8 U	**21**	6.9 U	**240**
LAI-13	10/4/2017	17 U	**200**	21 U	59 U	21 U	71 U	20 U	17 U	21 U	33 U	14 U	18 U	**15,000**	12 U
LAI-14	10/4/2017	**12**	**340**	2.1 U	**62**	**21**	**110**	**13**	**15**	**8.0**	3.3 U	**16**	**18**	**1,200**	1.2 U
LAI-15	10/4/2017	**8.8**	**480**	2.0 U	**41**	**9.4**	**71**	**10**	**16**	2.0 U	**18**	**13**	**16**	**9.0**	1.2 U
LAI-16	10/4/2017	**34**	**360**	**5.9**	**180**	**16**	**520**	**62**	**92**	**5.4**	**36**	**17**	**60**	**41**	1.1 U
LAI-23	10/6/2017	**200**	**530**	8.2 U	23 U	8.2 U	28 U	**110**	**250**	8.2 U	13 U	5.6 U	**18**	**30**	**4,200**
LAI-28	10/6/2017	**120**	**530**	2.1 U	**43**	**7.7**	**150**	**80**	**160**	2.1 U	3.2 U	1.4 U	**35**	2.6 U	**69**

Notes:

(a) MTCA Method B Screening Level

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

🟩 **Green Box** = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

µg/m^3 = micrograms per cubic meter

MTCA = Model Toxics Control Act

VOC = volatile organic compound

Table 7
C-23 Historical Data – Detected Constituents in Indoor Air
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	**VOCs**
	Analyte	Trichloroethene
	Unit of Measurement	µg/m^3
	Screening Level (a)	0.370
Location	Sample Date	
IA01-C23/IAR01C-23	11/20/2017	2.9 U
IA01-C23/IAR01C-23	12/11/2017	**0.50**
IA02-C23/IAR02C-23	11/20/2017	2.2 U
IA02-C23/IAR02C-23	12/11/2017	**0.40**
IA03-C23/IAR03-C23	11/20/2017	1.8 U
IA03-C23/IAR03-C23	12/11/2017	**0.55**
IA04-C23	11/20/2017	1.5 U
IA05-C23/IAR05C-23	11/20/2017	0.89 U
IA05-C23/IAR05C-23	12/11/2017	**0.96**
IA06-C23/IAR06-C23	11/20/2017	1.9 U
IA06-C23/IAR06-C23	12/11/2017	**0.42**
IAR04-C23	12/11/2017	**1.7**

Notes:

(a) MTCA Method B Screening Level

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

µg/m^3 = micrograms per cubic meter

MTCA = Model Toxics Control Act

VOC = volatile organic compound

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Table 8
C-29 / Former East Fuel Farm Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	TPH				Metals	VOCs						
			Analyte Unit of Measurement	Petroleum Hydrocarbons as Diesel mg/kg	Petroleum Hydrocarbons as Gasoline mg/kg	Petroleum Hydrocarbons as JP-A mg/kg	Total Petroleum Hydrocarbons mg/kg	Chromium, Total mg/kg	1,2,4-Trimethylbenzene µg/kg	1,3,5-Trimethylbenzene µg/kg	4-Isopropyltoluene µg/kg	Benzene µg/kg	cis-1,2-Dichloroethene µg/kg	Ethylbenzene µg/kg	Isopropylbenzene µg/kg
			Screening Level	2,000	30	2,000	30	42	--	800,000	--	0.277	5.15	343	8,000,000
Location	Sample Depth	Depth Units	Sample Date												
B14	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B14	6	FT	4/15/1994	25 U	**810**	**530**	--	--	--	--	--	**25**	--	**580**	--
B14	6	FT	4/15/1994	**29**	**113**	**210**	**61**	--	--	--	--	--	--	--	--
B14	6.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B14	15.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B14	22.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	10.5	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	3.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	5.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	12.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	15	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B15	20.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	37.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	6.5	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	11.5	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	13.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	15.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	20.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B17	27.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	6.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	8	FT	4/15/1994	**33**	25 U	25 U	**137**	--	--	--	--	--	--	--	--
B18	8.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	11.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	13.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	22.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B18	27.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	11.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	13.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	16.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	20.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--

Table 8
C-29 / Former East Fuel Farm Historical Data – Detected Constituents in Soil
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			Analyte Type	TPH				Metals	VOCs						
			Analyte	Petroleum Hydrocarbons as Diesel	Petroleum Hydrocarbons as Gasoline	Petroleum Hydrocarbons as JP-A	Total Petroleum Hydrocarbons	Chromium, Total	1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	4-Isopropyltoluene	Benzene	cis-1,2-Dichloroethene	Ethylbenzene	Isopropylbenzene
			Unit of Measurement	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	2,000	30	2,000	30	42	--	800,000	--	0.277	5.15	343	8,000,000
Location	Sample Depth	Depth Units	Sample Date												
B19	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	6.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	8.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	22.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B19	25.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B22	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B22	6.25	FT	4/15/1994	25 U	**1300**	**880**	--	--	--	--	--	**25**	--	**780**	--
B22	6.25	FT	4/15/1994	**18**	**376**	--	--	--	--	--	--	--	--	--	--
B22	6.25	FT	4/15/1994	**54**	**470**	**460**	**101**	--	--	--	--	--	--	--	--
B22	7.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
B22	10.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
C29-B3	1	FT	4/23/1996	--	--	--	--	**25**	--	--	--	--	--	--	--
C29-B3	7.5	FT	4/23/1996	--	--	--	--	**23**	--	--	--	--	--	--	--
C29-B3	10	FT	4/23/1996	--	--	--	--	**23**	--	--	--	--	--	--	--
C29-B3	15	FT	4/23/1996	--	--	--	--	**18**	--	--	--	--	--	--	--
C29-B4	1	FT	4/23/1996	--	--	--	--	**19**	--	--	--	--	--	--	--
C29-B4	2.5	FT	4/23/1996	--	--	--	--	**18**	--	--	--	--	--	--	--
C29-B4	7.5	FT	4/23/1996	--	--	--	--	**18**	--	--	--	--	--	--	--
C29-B4	15	FT	4/23/1996	--	--	--	--	**19**	--	--	--	--	--	--	--
C29-MW1	2.5	FT	4/19/1996	--	--	--	--	**21**	--	--	--	--	--	--	--
C29-MW1	5	FT	4/19/1996	--	--	--	--	**15**	--	--	--	--	--	--	--
C29-MW1	12.5	FT	4/19/1996	--	--	--	--	**19**	--	--	--	--	--	--	--
C29-MW1	15	FT	4/19/1996	--	--	--	--	**19**	400 U	400 U	400 U	--	400 U	400 U	400 U
C29-MW2	2.5	FT	4/19/1996	--	--	--	--	**20**	--	--	--	--	--	--	--
C29-MW2	5	FT	4/19/1996	--	--	--	--	**19**	--	--	--	--	--	--	--
C29-MW2	10	FT	4/19/1996	--	--	--	--	**17**	--	--	--	--	--	--	--
C29-MW2	15	FT	4/19/1996	--	--	--	--	**18**	--	--	--	--	--	--	--
C29-TP1	1.2	FT	4/11/1996	--	--	--	--	**24**	--	--	--	--	--	--	--
C29-TP1	2	FT	4/11/1996	--	--	--	--	**23**	--	--	--	--	--	--	--
C29-TP2	1.6	FT	4/11/1996	--	--	--	--	**19**	**14**	**6**	**0.84**	--	**0.3**	**0.47**	**0.69**
C29-TP2	3.5	FT	4/11/1996	--	--	--	--	**14**	**0.40**	0.20 U	0.20 U	--	**3.9**	0.20 U	0.20 U
C29-TP3	0.4	FT	4/11/1996	--	--	--	--	**16**	--	--	--	--	--	--	--
C29-TP3	2	FT	4/11/1996	--	--	--	--	**15**	--	--	--	--	--	--	--
C29-TP4	0.8	FT	4/11/1996	--	--	--	--	**18**	--	--	--	--	--	--	--
C29-TP4	1.8	FT	4/11/1996	--	--	--	--	**13**	--	--	--	--	--	--	--

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			Analyte Type	TPH				Metals	VOCs						
			Analyte	Petroleum Hydrocarbons as Diesel	Petroleum Hydrocarbons as Gasoline	Petroleum Hydrocarbons as JP-A	Total Petroleum Hydrocarbons	Chromium, Total	1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	4-Isopropyltoluene	Benzene	cis-1,2-Dichloroethene	Ethylbenzene	Isopropylbenzene
			Unit of Measurement	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	2,000	30	2,000	30	42	--	800,000	--	0.277	5.15	343	8,000,000
Location	Sample Depth	Depth Units	Sample Date												
C29-TP5A	0.5	FT	4/11/1996	--	--	--	--	**760**	--	--	--	--	--	--	--
C29-TP5A	2.6	FT	4/11/1996	--	--	--	--	**16**	--	--	--	--	--	--	--
C29-TP6	1.2	FT	4/11/1996	--	--	--	--	**20**	--	--	--	--	--	--	--
C29-TP7	1.4	FT	4/11/1996	--	--	--	--	**15**	--	--	--	--	--	--	--
C29-TP8	1	FT	4/11/1996	--	--	--	--	**25**	--	--	--	--	--	--	--
DW2	7	FT	12/12/2000	--	--	--	--	--	--	--	--	--	**46**	--	--
DW2	17	FT	12/12/2000	--	--	--	--	--	--	--	--	--	**87**	--	--
DW2	27	FT	12/12/2000	--	--	--	--	--	--	--	--	--	**200**	--	--
DW2	37	FT	12/12/2000	--	--	--	--	--	--	--	--	--	10 U	--	--
DW2	47	FT	12/12/2000	--	--	--	--	--	--	--	--	--	10 U	--	--
DW2	57	FT	12/12/2000	--	--	--	--	--	--	--	--	--	10 U	--	--
DW2	98.5	FT	12/12/2000	--	--	--	--	--	--	--	--	--	10 U	--	--
DW2	117	FT	12/12/2000	--	--	--	--	--	--	--	--	--	10 U	--	--
MW1	3.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW1	6.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW1	10	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW1	12.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW1	23.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	3.5	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	5.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	7.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	10.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	12.5	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	16.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW2	22.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW3	3.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW3	5.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW3	7.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW3	13.25	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
MW3	17.75	FT	4/15/1994	--	--	--	ND	--	--	--	--	--	--	--	--
TC-2	8.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-2	11.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-2	13.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	**64.5**	--	--
TC-2	16.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-2	18.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--

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			Analyte Type	TPH				Metals	VOCs						
			Analyte	Petroleum Hydrocarbons as Diesel	Petroleum Hydrocarbons as Gasoline	Petroleum Hydrocarbons as JP-A	Total Petroleum Hydrocarbons	Chromium, Total	1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	4-Isopropyltoluene	Benzene	cis-1,2-Dichloroethene	Ethylbenzene	Isopropylbenzene
			Unit of Measurement	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	2,000	30	2,000	30	42	--	800,000	--	0.277	5.15	343	8,000,000
Location	Sample Depth	Depth Units	Sample Date												
TC-2	23.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-3	8.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	**55**	--	--
TC-3	18.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-3	23.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--
TC-3	28.5	FT	12/20/1996	--	--	--	--	--	--	--	--	--	50 U	--	--

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Remedial Investigation Work Plan
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			Analyte Type	VOCs								
			Analyte	m-&p-Xylenes	n-Propylbenzene	o-Xylene	sec-Butylbenzene	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride	Xylenes, Total
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	150	8,000,000	232	8,000,000	273	32.5	0.206	0.009	831
Location	Sample Depth	Depth Units	Sample Date									
B14	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B14	6	FT	4/15/1994	--	--	--	--	25 U	--	--	--	**1,400**
B14	6	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B14	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B14	15.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B14	22.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	10.5	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	3.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	5.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	12.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	15	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B15	20.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	37.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	6.5	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	11.5	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	13.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	15.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	20.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B17	27.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	8	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	8.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	11.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	13.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	22.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B18	27.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	11.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	13.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	16.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	20.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--

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			Analyte Type	VOCs								
			Analyte	m-&p-Xylenes	n-Propylbenzene	o-Xylene	sec-Butylbenzene	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride	Xylenes, Total
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	150	8,000,000	232	8,000,000	273	32.5	0.206	0.009	831
Location	Sample Depth	Depth Units	Sample Date									
B19	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	8.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	22.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B19	25.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B22	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B22	6.25	FT	4/15/1994	--	--	--	--	25 U	--	--	--	**1,700**
B22	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B22	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B22	7.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
B22	10.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
C29-B3	1	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B3	7.5	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B3	10	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B3	15	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B4	1	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B4	2.5	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B4	7.5	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-B4	15	FT	4/23/1996	--	--	--	--	--	--	--	--	--
C29-MW1	2.5	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW1	5	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW1	12.5	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW1	15	FT	4/19/1996	400 U	400 U	400 U	400 U	400 U	--	**17,000**	400 U	--
C29-MW2	2.5	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW2	5	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW2	10	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-MW2	15	FT	4/19/1996	--	--	--	--	--	--	--	--	--
C29-TP1	1.2	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP1	2	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP2	1.6	FT	4/11/1996	**4.3**	**0.91**	**1.7**	**0.26**	**0.45**	--	0.20 U	0.20 U	--
C29-TP2	3.5	FT	4/11/1996	**0.48**	0.20 U	0.20 U	0.20 U	**0.32**	--	**0.85**	**0.33**	--
C29-TP3	0.4	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP3	2	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP4	0.8	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP4	1.8	FT	4/11/1996	--	--	--	--	--	--	--	--	--

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			Analyte	m-&p-Xylenes	n-Propylbenzene	o-Xylene	sec-Butylbenzene	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride	Xylenes, Total
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	150	8,000,000	232	8,000,000	273	32.5	0.206	0.009	831
Location	Sample Depth	Depth Units	Sample Date									
C29-TP5A	0.5	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP5A	2.6	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP6	1.2	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP7	1.4	FT	4/11/1996	--	--	--	--	--	--	--	--	--
C29-TP8	1	FT	4/11/1996	--	--	--	--	--	--	--	--	--
DW2	7	FT	12/12/2000	--	--	--	--	--	**12**	**380**	--	--
DW2	17	FT	12/12/2000	--	--	--	--	--	10 U	**120**	--	--
DW2	27	FT	12/12/2000	--	--	--	--	--	10 U	**480**	--	--
DW2	37	FT	12/12/2000	--	--	--	--	--	10 U	10 U	--	--
DW2	47	FT	12/12/2000	--	--	--	--	--	10 U	10 U	--	--
DW2	57	FT	12/12/2000	--	--	--	--	--	10 U	10 U	--	--
DW2	98.5	FT	12/12/2000	--	--	--	--	--	10 U	10 U	--	--
DW2	117	FT	12/12/2000	--	--	--	--	--	10 U	10 U	--	--
MW1	3.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW1	6.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW1	10	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW1	12.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW1	23.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	3.5	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	5.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	7.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	10.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	12.5	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	16.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW2	22.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW3	3.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW3	5.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW3	7.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW3	13.25	FT	4/15/1994	--	--	--	--	--	--	--	--	--
MW3	17.75	FT	4/15/1994	--	--	--	--	--	--	--	--	--
TC-2	8.5	FT	12/20/1996	--	--	--	--	--	--	**972**	--	--
TC-2	11.5	FT	12/20/1996	--	--	--	--	--	--	**705**	--	--
TC-2	13.5	FT	12/20/1996	--	--	--	--	--	--	**3,360**	--	--
TC-2	16.5	FT	12/20/1996	--	--	--	--	--	--	**1,940**	--	--
TC-2	18.5	FT	12/20/1996	--	--	--	--	--	--	**1,430**	--	--

Table 8
C-29 / Former East Fuel Farm Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type		VOCs							
			Analyte	m-&p-Xylenes	n-Propylbenzene	o-Xylene	sec-Butylbenzene	Toluene	trans-1,2-Dichloroethene	Trichloroethene	Vinyl Chloride	Xylenes, Total
			Unit of Measurement	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
			Screening Level	150	8,000,000	232	8,000,000	273	32.5	0.206	0.009	831
Location	Sample Depth	Depth Units	Sample Date									
TC-2	23.5	FT	12/20/1996	--	--	--	--	--	--	**173**	--	--
TC-3	8.5	FT	12/20/1996	--	--	--	--	--	--	**1,430**	--	--
TC-3	18.5	FT	12/20/1996	--	--	--	--	--	--	50 U	--	--
TC-3	23.5	FT	12/20/1996	--	--	--	--	--	--	50 U	--	--
TC-3	28.5	FT	12/20/1996	--	--	--	--	--	--	50 U	--	--

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation lin

Bold = detected compound

Green Box = detected concentration is greater than preliminary screening level

-- = not analyzed

Abbreviations and Acronyms:

DRO - diesel-range organics
FT = feet
µg/kg = micrograms per kilogram
mg/kg = milligrams per kilogram
ND = not detected
ORO = oil-range organics
TPH = total petroleum hydrocarbons
VOC = volatile organic compound

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Table 9
C-29 / Former East Fuel Farm Historical Data – Detected Constituents in Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	TPH			Metals				SVOCs		VOCs				
	Analyte	Petroleum Hydrocarbons as Diesel	Petroleum Hydrocarbons as Jet-A	Gasoline	Chromium, Total	Chromium, Dissolved	Copper	Zinc	4-Nitrophenol	Phenol	1,1,1-Trichloroethane	1,1-Dichloroethene	1,2,4-Trimethylbenzene	1,2-Dichloroethane	1,2-Dichloropropane
	Unit of Measurement	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
	Screening Level	500	500	800	100	100	640	4,800		2,400	200	7	--	0.481	1.22
Location	Sample Date														
AF-1	1/5/1996	--	--	--	**55,000 J**	--	**34**	**58**	**16 J**	**56**	--	--	**2.6**	--	--
C29-MW1	5/7/1996	--	--	--	10 U	--	--	--	--	--	--	5 U	--	--	--
C29-MW1	2/24/1999	--	--	--	10 U	10 U	--	--	--	--	--	--	--	**13**	--
C29-MW2	5/7/1999	--	--	--	10 U	--	--	--	--	--	--	--	--	--	--
DW2	12/28/1999	--	--	--	--	--	--	--	--	--	**49**	--	--	**26**	**10**
DW2	3/8/2000	--	--	--	--	--	--	--	--	--	**45**	--	--	**15**	**13**
HMB1	2/24/1999	--	--	--	10 U	10 U	--	--	--	--	--	5 U	--	5 U	--
MW1	4/27/1994	1,000 U	1,000 U	**1,000**	--	--	--	--	--	--	--	--	--	--	--
MW1	5/7/1996	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW1	2/24/1998	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW1	2/24/1999	--	--	--	--	--	--	--	--	--	--	**34**	--	5 U	--
MW1	10/24/2001	**320**	--	**50**	--	--	--	--	--	--	--	--	--	--	--
MW2	4/27/1994	1,000 U	**1,000**	**2,000**	--	--	--	--	--	--	--	--	--	--	--
MW2	5/7/1996	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW2	2/24/1998	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW2	2/24/1999	--	--	--	--	--	--	--	--	--	--	**9**	--	**8**	--
MW2	10/24/2001	**150**	--	**55**	--	--	--	--	--	--	--	--	--	--	--
MW3	4/27/1994	1,000 U	**1,300**	1,000 U	--	--	--	--	--	--	--	--	--	--	--
MW3	5/7/1996	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW3	2/24/1998	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW3	2/24/1999	--	--	--	--	--	--	--	--	--	--	**120**	--	**180**	--
MW3	10/24/2001	**380**	--	50 U	--	--	--	--	--	--	--	--	--	--	--
MW4	4/27/1994	1,000 U	**1,100**	**2,000**	**330**	--	**320**	--	--	--	--	--	--	--	--
MW4	5/7/1996	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW4	2/24/1998	--	--	--	10 U	10 U	--	--	--	--	--	--	--	--	--
MW4	2/24/1999	--	--	--	--	--	--	--	--	--	--	5 U	--	5 U	--
MW4	10/24/2001	**990**	--	**1,100**	--	--	--	--	--	--	--	--	--	--	--
SCPWD-1	2/24/1999	--	--	--	10 U	10 U	--	--	--	--	--	**45**	--	5 U	--

Table 9
C-29 / Former East Fuel Farm Historical Data – Detected Constituents in Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs													VOCs; SVOCs
	Analyte Unit of Measurement	1,3,5-Trimethylbenzene µg/L	4-Isopropyltoluene µg/L	Benzene µg/L	Chloroethane µg/L	cis-1,2-Dichloroethene µg/L	Ethylbenzene µg/L	m-&p-Xylenes µg/L	o-Xylene µg/L	Toluene µg/L	trans-1,2-Dichloroethene µg/L	Trichloroethene µg/L	Vinyl Chloride µg/L	Xylenes, Total µg/L	Naphthalene µg/L
	Screening Level	80	--	0.795	--	16	700	310	440	640	100	0.54	0.2	1,600	160
Location	Sample Date														
AF-1	1/5/1996	**2.5**	**2.0**	--	--	**13**	--	**1.1**	**1.1**	**4.8**	--	**31**	**4.8**	--	**2.4**
C29-MW1	5/7/1996	--	--	--	--	**21,000**	--	--	--	--	800 U	**74,700**	800 U	--	--
C29-MW1	2/24/1999	--	--	--	**27**	**26,000**	--	--	--	--	**490**	**18,000**	**80**	--	--
C29-MW2	5/7/1999	--	--	--	--	**956**	--	--	--	--	80 U	**10,400**	80 U	--	--
DW2	12/28/1999	--	--	--	--	5 U	--	--	--	--	--	5 U	--	--	--
DW2	3/8/2000	--	--	--	--	5 U	--	--	--	--	--	5 U	--	--	--
HMB1	2/24/1999	--	--	--	5 U	5 U	--	--	--	--	5 U	5 U	5 U	--	--
MW1	4/27/1994	--	--	**7.8**	--	--	**2**	--	--	**3.7**	--	--	--	**1.8**	--
MW1	5/7/1996	--	--	--	--	**3,730**	--	--	--	--	**189**	**1,490**	80 U	--	--
MW1	2/24/1998	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW1	2/24/1999	--	--	--	5 U	**6,700**	--	--	--	--	**460**	**4,400**	**85**	--	--
MW1	10/24/2001	--	--	**33**	--	--	10 U	--	--	10 U	--	--	--	30 U	--
MW2	4/27/1994	--	--	**13**	--	--	**88**	--	--	**47**	--	--	--	**470**	--
MW2	5/7/1996	--	--	--	--	**301**	--	--	--	--	8 U	**33.9**	**131**	--	--
MW2	2/24/1998	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW2	2/24/1999	--	--	--	5 U	**2,600**	--	--	--	--	**50**	**79**	**920**	--	--
MW2	10/24/2001	--	--	**12**	--	--	**5**	--	--	**2**	--	--	--	**4**	--
MW3	4/27/1994	--	--	**65**	--	--	**0.6**	--	--	**3.2**	--	--	--	**1.8**	--
MW3	5/7/1996	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW3	2/24/1998	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW3	2/24/1999	--	--	--	5 U	**9,400**	--	--	--	--	**530**	**7,900**	**440**	--	--
MW3	10/24/2001	--	--	**42**	--	--	10 U	--	--	10 U	--	--	--	30 U	--
MW4	4/27/1994	--	--	**130**	--	--	**75**	--	--	**8.7**	--	--	--	**80**	--
MW4	5/7/1996	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW4	2/24/1998	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MW4	2/24/1999	--	--	--	5 U	**83**	--	--	--	--	**58**	**19**	**1,100**	--	--
MW4	10/24/2001	--	--	**160**	--	--	**96**	--	--	10 U	--	--	--	130 U	--
SCPWD-1	2/24/1999	--	--	--	5 U	**7,400**	--	--	--	--	**79**	**17,000**	**880**	--	--

Notes:

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

Green Box = detected concentration is greater than preliminary screening level

-- = not analyzed

Abbreviations and Acronyms:

µg/L = micrograms per liter

SVOC = semivolatile organic compound

TPH = total petroleum hydrocarbons

VOC = volatile organic compound

Table 10
Deep Aquifer Historical Data – Detected Constituents in Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

			Analyte Type	VOCs		
			Analyte	cis-1,2-Dichloroethene	trans-1,2-Dichloroethene	Trichloroethene
			Unit of Measurement	mg/kg	mg/kg	mg/kg
			Screening Level	0.00515	0.0325	0.000206
Location	Sample Depth	Depth Units	Sample Date			
DW1	57.5	FT	12/12/2000	0.01 U	0.01 U	**0.01**
DW1	77	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW1	97.5	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW1	117	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW1	137	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW2	7	FT	12/12/2000	**0.046**	**0.012**	**0.38**
DW2	17	FT	12/12/2000	**0.087**	0.01 U	**0.12**
DW2	27	FT	12/12/2000	**0.2**	0.01 U	**0.48**
DW2	37	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW2	47	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW2	57	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW2	98.5	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW2	117	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	7	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	36	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	66	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	76	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	136	FT	12/12/2000	0.01 U	0.01 U	0.01 U
DW3	151	FT	12/12/2000	0.01 U	0.01 U	0.01 U

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

FT = feet

mg/kg = milligrams per kilogram

VOC = volatile organic compound

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Table 11
Deep Aquifer Historical Data – Detected Constituents in Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

	Analyte Type	VOCs				
	Analyte	1,1,1-Trichloroethane	1,2-Dichloroethane	1,2-Dichloropropane	cis-1,2-Dichloroethene	Trichloroethene
	Unit of Measurement	µg/L	µg/L	µg/L	µg/L	µg/L
	Screening Level	200	0.481	1.22	16.0	0.540
Location	Sample Date					
DW1	12/28/1999	5 U	5 U	5 U	5 U	**8**
DW1	3/8/2000	5 U	5 U	5 U	**5**	**62**
DW1	10/17/2003	5 U	5 U	5 U	5 U	**81**
DW2	12/28/1999	**49**	**26**	**10**	5 U	5 U
DW2	3/8/2000	**45**	**15**	**13**	5 U	5 U
DW3	5/19/2000	5 U	5 U	5 U	5 U	5 U

Notes:

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

Bold = detected compound

Green Box = detected concentration is greater than preliminary screening level

Abbreviations and Acronyms:

µg/L = micrograms per liter

VOC = volatile organic compound

Table 12
Screening Levels for Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

CAS	Analyte Group	Analyte	Units of Measurement	Protection of Groundwater Saturated @ 13 degrees Celsius or Vapor Intrusion	MTCA Method A (Lead, Mercury, TPH Only)	Direct Contact Pathway (Ingestion Only) Use Method B Formula Values		Preliminary Screening Levels (Before adjustment for Background)	Background Soil Metals Conc. Puget Sound Region 90th Percentile Value	Adjusted Screening Levels
						Non-carcinogen	Carcinogen			
877-24-7	Conventionals	Total Organic Carbon	mg/kg							
7440-38-2	Metals	Arsenic	mg/kg	0.146		24	0.667	0.146	7	7
7440-43-9	Metals	Cadmium	mg/kg	0.0349		80		0.0349	1	1
7440-47-3	Metals	chromium (total)	mg/kg					42	42	42
16065-83-1	Metals	chromium(III)	mg/kg	100		120,000		100		100
18540-29-9	Metals	chromium(VI)	mg/kg	0.926		240		0.926		0.926
7439-92-1	Metals	Lead	mg/kg	150	250			150	17	150
7439-97-6	Metals	Mercury	mg/kg	0.105	2.0			0.105	0.07	0.105
12674-11-2	PCBs	Aroclor 1016	mg/kg	*		5.6	14.3	5.6		5.6
11104-28-2	PCBs	Aroclor 1221	mg/kg							
11141-16-5	PCBs	Aroclor 1232	mg/kg							
53469-21-9	PCBs	Aroclor 1242	mg/kg							
12672-29-6	PCBs	Aroclor 1248	mg/kg							
11097-69-1	PCBs	Aroclor 1254	mg/kg	*		1.6	0.5	0.5		0.5
11096-82-5	PCBs	Aroclor 1260	mg/kg	*			0.5	0.5		0.5
11100-14-4	PCBs	Aroclor 1268	mg/kg							
1336-36-3	PCBs	Total PCBs	mg/kg	*			0.5	0.5		0.5
PHC_DIESEL	TPH	TPH, diesel-range organics	mg/kg		2,000			2,000		2,000
PHC_MOIL	TPH	TPH, heavy oils	mg/kg		2,000			2,000		2,000
PHC_GAS_Benz	TPH	TPH: gasoline-range organics, benzene present*	mg/kg		30			30		30
PHC_GAS_only	TPH	TPH: gasoline-range organics, no detectable benzene*	mg/kg		100			100		100
630-20-6	VOCs	1,1,1,2-Tetrachloroethane	µg/kg	*		2,400,000	38,500	38,500		38,500
79-34-5	VOCs	1,1,2,2-Tetrachloroethane	µg/kg	0.080		1,600,000	5,000	0.080		0.080
79-00-5	VOCs	1,1,2-Trichloroethane	µg/kg	0.278		320,000	17,600	0.278		0.278
107-06-2	VOCs	1,2-Dichloroethane	µg/kg	1.56		480,000	11,000	1.56		1.56
78-87-5	VOCs	1,2-Dichloropropane	µg/kg	1.67		7,200,000	27,800	1.67		1.67
591-78-6	VOCs	2-Hexanone	µg/kg							
99-87-6	VOCs	4-Isopropyltoluene	µg/kg							
108-10-1	VOCs	4-Methyl-2-Pentanone	µg/kg			6,400,000		6,400,000		6,400,000
67-64-1	VOCs	acetone	µg/kg	2,070		72,000,000		2,070		2,070
71-43-2	VOCs	benzene	µg/kg	0.277		320,000	18,200	0.277		0.277
75-15-0	VOCs	carbon disulfide	µg/kg	266		8,000,000		266		266
56-23-5	VOCs	Carbon Tetrachloride	µg/kg	0.274		320,000	14,300	0.274		0.274
75-00-3	VOCs	Chloroethane	µg/kg	*						
67-66-3	VOCs	Chloroform	µg/kg	0.479		800,000	32,300	0.479		0.479
98-82-8	VOCs	cumene (common name for isopropylbenzene)	µg/kg	*		8,000,000		8,000,000		8,000,000
75-34-3	VOCs	dichloroethane;1,1-	µg/kg	2.61		16,000,000	175,000	2.61		2.61
75-35-4	VOCs	dichloroethylene;1,1-	µg/kg	2.46		4,000,000		2.46		2.46

Table 12
Screening Levels for Soil
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

CAS	Analyte Group	Analyte	Units of Measurement	Protection of Groundwater Saturated @ 13 degrees Celsius or Vapor Intrusion	MTCA Method A (Lead, Mercury, TPH Only)	Direct Contact Pathway (Ingestion Only) Use Method B Formula Values		Preliminary Screening Levels (Before adjustment for Background)	Background Soil Metals Conc. Puget Sound Region 90th Percentile Value	Adjusted Screening Levels
						Non-carcinogen	Carcinogen			
156-59-2	VOCs	dichloroethylene;1,2-,cis	µg/kg	5.15		160,000		5.15		5.15
156-60-5	VOCs	dichloroethylene;1,2-,trans	µg/kg	32.5		1,600,000		32.5		32.5
100-41-4	VOCs	ethylbenzene	µg/kg	343		8,000,000		343		343
106-93-4	VOCs	Ethylene Dibromide (1,2-Dibromoethane)	µg/kg	*		720,000	500	500		500
78-93-3	VOCs	methyl ethyl ketone	µg/kg			48,000,000		48,000,000		48,000,000
1634-04-4	VOCs	Methyl T-Butyl Ether	µg/kg	7.23			556,000	7.23		7.23
75-09-2	VOCs	methylene chloride	µg/kg	1.48		480,000	500,000	1.48		1.48
91-20-3	VOCs	Naphthalene	µg/kg	236		1,600,000		236		236
103-65-1	VOCs	propylbenzene;n-	µg/kg			8,000,000		8,000,000		8,000,000
135-98-8	VOCs	sec-butylbenzene	µg/kg			8,000,000		8,000,000		8,000,000
127-18-4	VOCs	tetrachloroethylene (PCE)	µg/kg	2.76		480,000	476,000	2.76		2.76
108-88-3	VOCs	toluene	µg/kg	273		6,400,000		273		273
1330-20-7	VOCs	Total xylenes	µg/kg	831		16,000,000		831		831
71-55-6	VOCs	trichloroethane;1,1,1-	µg/kg	84.3		160,000,000		84.3		84.3
79-01-6	VOCs	trichloroethylene (TCE)	µg/kg	0.206		40,000	12,000	0.206		0.206
95-63-6	VOCs	trimethylbenzene;1,2,4-	µg/kg	*						
108-67-8	VOCs	trimethylbenzene;1,3,5-	µg/kg			800,000		800,000		800,000
75-01-4	VOCs	vinyl chloride	µg/kg	0.009		240,000	667	0.009		0.009

Notes:

Soil screening levels have not been adjusted for PQL because the achieveable laboratory reporting limits are based on dry weight. PQL adjustments will be presented in the RI.

*No partitioning information provided in CLARC for protection of groundwater calculation

Abbreviations and Acronyms:

CAS = Chemical Abstract Service
µg/kg = micrograms per kilogram
mg/kg =milligrams per kilogram
MTCA = Model Toxics Control Act
PCB = polychlorinated biphenyl
TPH = total petroleum hydrocarbons
VOC = volatile organic compound

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Table 13
Screening Levels for Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

CAS	Analyte Group	Analyte	Unit of Measurement	MCL	Washington State MCL	MTCA Method A (Lead, Mercury, and TPH Only)	Method B Standard Formula Values		Groundwater Protective of Vapor Intrusion (a)	Preliminary Screening Levels (Before adjustment for PQL and Background)	Laboratory PQL	Groundwater Background Concentration	Adjusted Screening Level
							Non-carcinogen	Carcinogen					
NA	Conventionals	Ferrous Iron	µg/L							NA	--		
14797-55-8	Conventionals	Nitrate	µg/L	10,000	10,000		25,600			10,000	153		10,000
14808-79-8	Conventionals	Sulfate	µg/L							NA	260		
877-24-7	Conventionals	Total Organic Carbon	µg/L							NA	500		
74-84-0	Dissolved Gases	Ethane	µg/L							NA	10		
74-85-1	Dissolved Gases	Ethene	µg/L							NA	10		
74-82-8	Dissolved Gases	Methane	µg/L							NA	10		
7440-38-2	Metals	Arsenic	µg/L	10	10		4.8	0.058		0.058	1	5	5
7440-43-9	Metals	Cadmium	µg/L	5	5		8			5	1		5
7440-47-3	Metals	chromium (total)	µg/L	100	100					100	2		100
16065-83-1	Metals	chromium(III)	µg/L		100		24,000			100	10		100
18540-29-9	Metals	chromium(VI)	µg/L				48			48	10		48
7439-92-1	Metals	Lead	µg/L	15	15	15				15	1		15
7439-97-6	Metals	Mercury	µg/L	2	2	2				2	0.2		2
PHC_DIESEL	TPH	TPH, diesel-range organics	µg/L			500				500	130		500
PHC_MOIL	TPH	TPH, heavy oils	µg/L			500				500	250		500
PHC_GAS_Benz	TPH	TPH: gasoline-range organics, benzene present*	µg/L			800				800	50		800
PHC_GAS_only	TPH	TPH: gasoline-range organics, no detectable benzene	µg/L			1,000				1,000	50		1,000
123-91-1	SVOCs	1,4-Dioxane	µg/L				240	0.44		0.44	0.40		0.44
630-20-6	VOCs	1,1,1,2-Tetrachloroethane	µg/L				240	1.68		1.68	0.5		1.68
79-34-5	VOCs	1,1,2,2-Tetrachloroethane	µg/L				160	0.219		0.219	0.5		0.50
79-00-5	VOCs	1,1,2-Trichloroethane	µg/L	5	5		32	0.768		0.768	0.5		0.768
591-78-6	VOCs	2-Hexanone	µg/L							NA	10		
99-87-6	VOCs	4-Isopropyltoluene	µg/L							NA	2		
108-10-1	VOCs	4-Methyl-2-Pentanone	µg/L				640			640	10		640
67-64-1	VOCs	acetone	µg/L				7,200			7,200	25		7,200
71-43-2	VOCs	benzene	µg/L	5	5		32	0.795	2.40	0.795	0.5		0.795
75-15-0	VOCs	Carbon Disulfide	µg/L				800			800	2		800
56-23-5	VOCs	Carbon Tetrachloride	µg/L	5	5		32	0.625		0.625	0.5		0.625
75-00-3	VOCs	Chloroethane	µg/L							NA	2		
67-66-3	VOCs	Chloroform	µg/L	80	80		80	1.41		1.41	0.5		1.41
98-82-8	VOCs	cumene (common name for isopropylbenzene)	µg/L				800			800	2		800
75-34-3	VOCs	dichloroethane;1,1-	µg/L				1,600	7.68	11.2	7.68	2		7.68
107-06-2	VOCs	dichloroethane;1,2-	µg/L	5	5		48	0.481		0.481	0.02		0.481
75-35-4	VOCs	dichloroethylene;1,1-	µg/L	7	7		400			7	2		7
156-59-2	VOCs	dichloroethylene;1,2-,cis	µg/L	70	70		16			16	2		16
156-60-5	VOCs	dichloroethylene;1,2-,trans	µg/L	100	100		160			100	2		100
78-87-5	VOCs	dichloropropane;1,2-	µg/L	5	5		720	1.22		1.22	0.5		1.22
100-41-4	VOCs	ethylbenzene	µg/L	700	700		800			700	2		700
106-93-4	VOCs	Ethylene Dibromide (1,2-Dibromoethane)	µg/L	0.05	0.05		72	0.02		0.02	0.01		0.02

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Table 13
Screening Levels for Groundwater
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

CAS	Analyte Group	Analyte	Unit of Measurement	MCL	Washington State MCL	MTCA Method A (Lead, Mercury, and TPH Only)	Method B Standard Formula Values		Groundwater Protective of Vapor Intrusion (a)	Preliminary Screening Levels (Before adjustment for PQL and Background)	Laboratory PQL	Groundwater Background Concentration	Adjusted Screening Level
							Non-carcinogen	Carcinogen					
78-93-3	VOCs	methyl ethyl ketone	µg/L				4,800			4,800	10		4,800
1634-04-4	VOCs	Methyl T-Butyl Ether	µg/L					24.3		24.3	2		24.3
75-09-2	VOCs	Methylene Chloride	µg/L	5	5		48	21.9		5	5		5
91-20-3	VOCs	naphthalene	µg/L				160			160	2		160
103-65-1	VOCs	n-Propylbenzene	µg/L				800			800	2		800
135-98-8	VOCs	sec-Butylbenzene	µg/L				800			800	2		800
127-18-4	VOCs	tetrachloroethylene (PCE)	µg/L	5	5		48	20.8	22.9	5	2		5
108-88-3	VOCs	toluene	µg/L	1,000	1,000		640			640	2		640
1330-20-7	VOCs	Total xylenes	µg/L	10,000	10,000		1,600			1,600	4		1,600
71-55-6	VOCs	trichloroethane;1,1,1-	µg/L	200	200		16,000		5,240	200	2		200
79-01-6	VOCs	trichloroethylene (TCE)	µg/L	5	5		4	0.54	1.55	0.54	0.5		0.54
95-63-6	VOCs	trimethylbenzene;1,2,4-	µg/L							NA	2		
108-67-8	VOCs	trimethylbenzene;1,3,5-	µg/L				80			80	2		80
75-01-4	VOCs	vinyl chloride	µg/L	2	2		24	0.029	0.347	0.029	0.02		0.029

Notes:
(a) Values provide only for constituents on the Air analyte list

Abbreviations/Acronyms:
CAS = Chemical Abstract Service
µg/L = micrograms per liter
MCL = maximum contaminant level
MTCA = Model Toxics Control Act
NA = not available
PQL = practical quantitation limit
SVOC = semivolatile organic compound
TPH = total petroleum hydrocarbons
VOC = volatile organic compound

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Table 14
Screening Levels for Soil Gas
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

CAS	Analyte	Indoor Air Screening Level (µg/m^3)			Shallow Soil Gas Screening Level (µg/m^3)			Groundwater Screening Level (µg/L)		
		Carcinogenic	Non-carcinogenic	Preliminary Screening Levels (µg/m^3)	Carcinogenic	Non-carcinogenic	Preliminary Screening Levels (µg/m^3)	Carcinogenic	Non-carcinogenic	Preliminary Screening Levels (µg/L)
71-43-2	benzene	0.321	13.7	0.321	10.7	457	10.7	2.4	103	2.4
75-34-3	dichloroethane;1,1-	1.56		1.56	52.1		52.1	11.2		11.2
127-18-4	tetrachloroethylene	9.62	18.3	9.62	321	610	321	22.9	43.5	22.9
71-55-6	trichloroethane;1,1,1-		2,290	2,290		76,200	76,200		5,240	5,240
79-01-6	trichloroethylene	0.37	0.914	0.37	12.3	30.5	12.3	1.55	3.84	1.55
75-01-4	vinyl chloride	0.28	45.7	0.28	9.33	1,524	9.33	0.347	56.7	0.347

Notes:

Analyte list was determined based on results from TO-15 analysis of soil gas conducted during the 2017 and 2018 Phase II investigation by LAI.

Abbreviations/Acronyms:

CAS = Chemical Abstract Service
µg/L = microgram per liter
µg/m^3 = microgram per cubic meter

Table 15
Existing Monitoring Wells
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Well Type	Well ID	Location	DTB (ft)	DTW (ft)	Condition of Well - July 2018	Depth to Sediment	Water Description	Recommended Rehab Measures
Shallow Monitoring Well	TRM-MW-1	East Fuel Farm	23.2	10	Unsecured, no lock, well cap is labeled	Soft at bottom	Clear/no odor	Redevelop, replace well cap, replace lock
	TRM-MW-2	East Fuel Farm	16.9	7.8	Secured, buried by gravel, some water inside monument	No sediment	Clear	Redevelop, regrade around well or extend monument, replace well cap, replace lock
	TRM-MW-3	East Fuel Farm	17.6	6.4	Secured-no lock	No sediment	Slight petroleum-like odor	Redevelop, replace lock
	TRM-MW-4	East Fuel Farm	16.2	8.2	Secured-no lock	15	Gray/faint petroleum-like odor	Redevelop, replace lock
	TRM-HMB-1	East Fuel Farm	19.4	NM	Secured-no lock	18.3	Clear/no odor	Redevelop, replace bolts on monument, replace lock
	C-29-MW-1	Former Building C-29	19	NM	Secured, monument destoyed	No sediment	Clear/no odor	Redevelop, replace monument
	C29-MW-2	Former Building C-29	14.35	5	Secured, some water inside monument		Clear/unknown odor	Redevelop
	SCPWD-1	Former Building C-29	19.1	5.3	Unsecured, cap loose, sediment in monument, sediment in bottom of well	18.6	No odor	Redevelop, replace well cap, remove sediment from monument
	SCPWD-2	Building C-19	NM	NM	Did not open, appears in good condition	N/A	N/A	Redevelop
	SCPWD-3	Building C-19	NM	NM	No monument lid, cap is exposed, did not remove sediment that filled monument to protect integrity of PVC well casing	N/A	N/A	Redevelop, replace monument lid or monument as necessary, replace well cap, replace lock
	SCPWD-4	Building C-19	NM	NM	Did not open, monument at a ~45 deg angle, potential damage to casing	N/A	N/A	Redevelop, inspect casing integrity, replace monument
Deep Monitoring Well	DW1	Building C-19	NM	NM	Buried under asphalt (marked estimated location with paint)	N/A	N/A	Uncover well, extend monument to paved surface, re-develop
	DW2	East Fuel Farm	NM	NM	Secured, cap is labeled	N/A	Brown thick water	Redevelop
	DW3	NE of Building C-19	NM	NM	Did not open, appears in good condition	N/A	N/A	Redevelop

Abbreviations and Acronyms:

N/A =	not applicable
NM =	not measured
DTB =	depth to bottom of well casing from top of casing
DTW =	depth to water from top of casing
ft =	feet

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Table 16
Summary of Remedial Investigation Sampling and Analyses
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

		Soil									Groundwater							Soil Gas/ Sub Slab	Selection Rationale
Exploration ID	Investigation Area	# of Samples	VOCs	DRO / ORO	GRO	Metals	PCBs	TOC	Grain Size	TAT	VOCs	DRO / ORO	GRO	Dis. Metals	1,4-Dioxane	NA Params	TAT	VOCs	
SCPWD-2	C-19										X						Std		Existing Well
SCPWD-3	C-19										X				X	X (c)	Std		Existing Well
SCPWD-4	C-19										X				X	X (c)	Std		Existing Well
SCPWD-1	C-29/Fuel Farm										X	X	X	X (a)		X (c)	Std		Existing Well
MW-1	C-29/Fuel Farm										X	X	X	X (b)			Std		Existing Well
MW-2	C-29/Fuel Farm										X	X	X	X (b)		X (c)	Std		Existing Well
MW-3	C-29/Fuel Farm										X	X	X	X (a)			Std		Existing Well
MW-4	C-29/Fuel Farm										X	X	X	X (a)		X (c)	Std		Existing Well
HMB1	C-29/Fuel Farm										X	X	X	X (a)			Std		Existing Well
C29-MW1	C-29/Fuel Farm										X	X	X	X (a)		X (c)	Std		Existing Well
C29-MW2	C-29/Fuel Farm										X	X	X	X (b)			Std		Existing Well
DW1	Deep Aquifer										X				X	X (c)	Std		Existing Deep Well
DW2	Deep Aquifer										X				X	X (c)	Std		Existing Deep Well
DW3	Deep Aquifer										X					X (c)	Std		Existing Deep Well
RISB-01	C-19	1-3 (f)	X							Std	X						3-Day		Delineate contamination at GP12 and GP19 on the east and south along property line. No soils data.
RISB-02	C-19	1-3 (f)	X							Std	X						3-Day		Delineate contamination at GP12 and GP19 on the east and south along property line. No soils data.
RISB-03	C-19	1-3 (f)	X	X (***)		X (**)(d)				Std	X	X (*)		X (a)			3-Day		Delineate north and east sides of degreaser pit.
RISB-04	C-19	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)		X (b)			3-Day	X	Delineate north and east sides of degreaser pit.
RISB-05	C-19	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)		X (b)			3-Day	X	Delineate north and east sides of degreaser pit.
RISB-06	C-19	1-3 (f)	X	X (***)		X (**)(d)				Std	X	X (*)		X (a)			3-Day		Former drum storage area with no previous sampling.
RISB-07	C-19	1-3 (f)	X	X (*)				X	X	Std	X	X (*)		X (a)			3-Day		Adjacent to building in area with no previous sampling.
RISB-08	C-19	1-3 (f)	X							Std	X						3-Day		Further delineate around the west, southwest, and southern side of the VOC contamination off the south corner of C-19
RISB-09	C-19	1-3 (f)	X							Std	X						3-Day		Further delineate around the west, southwest, and southern side of the VOC contamination off the south corner of C-19
RISB-10	C-19	1-3 (f)	X							Std	X						3-Day		Further delineate around the west, southwest, and southern side of the VOC contamination off the south corner of C-19
RISB-11	C-19	1-3 (f)	X	X (*)		X (**)(d)		X	X	Std									Further delineate around the west, southwest, and southern side of the VOC contamination off the south corner of C-19
RISB-54	C-19	1-3 (f)	X							Std	X (i)						Std	X	Delineation of degreaser pit source area
RISB-55	C-19	1-3 (f)	X							Std	X (i)						Std	X	Delineation of degreaser pit source area
RISB-12	C-20, -21, -22	1-3 (f)	X	X (***)			X (*)			Std								TBD	Delineate vertical extent of contamination at LAI-12
RISB-13	C-20, -21, -22	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)		X (a)			3-Day	TBD	Delineate vertical extent of contamination at LAI-20
RISB-14	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Delineate extent of contamination and investigate source on the north side of C-21 where moderate levels of TCE were detected in soil gas
RISB-15	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Delineate extent of contamination and investigate source on the north side of C-21 where moderate levels of TCE were detected in soil gas
RISB-16	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Further investigation on north side of C-22 where high levels of TCE were detected in soil gas; investigate source

Table 16
Summary of Remedial Investigation Sampling and Analyses
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

		Soil									Groundwater							Soil Gas/ Sub Slab	Selection Rationale
Exploration ID	Investigation Area	# of Samples	VOCs	DRO / ORO	GRO	Metals	PCBs	TOC	Grain Size	TAT	VOCs	DRO / ORO	GRO	Dis. Metals	1,4-Dioxane	NA Params	TAT	VOCs	
RISB-17	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Further investigation on north side of C-22 where high levels of TCE were detected in soil gas; investigate source
RISB-18	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil
RISB-19	C-20, -21, -22	1-3 (f)	X	X (*)				X	X	Std	X	X (*)		X (a)			3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil
RISB-20	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil
RISB-21	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)		X (a)			3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil
RISB-22	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil
RISB-23	C-20, -21, -22	1-3 (f)	X	X (*)						Std	X	X (*)					3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-24	C-20, -21, -22	1-3 (f)	X	X (***)			X (*)			Std	X	X (*)					3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-25	C-20, -21, -22	1-3 (f)	X	X (***)			X (*)			Std	X	X (*)					3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-26	C-20, -21, -22	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)		X (a)			3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-27	C-20, -21, -22	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)		X (a)			3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-28	C-20, -21, -22	1-3 (f)	X	X (***)		X (**)(d)	X (*)	X	X	Std	X	X (*)		X (a)			3-Day	TBD	Delineation of contamination and source evaulation based on results from LAI-10, -20, and - 27
RISB-49	C-20, -21, -22	1-3 (f)	X	X (***)			X (*)			Std	X	X (*)		X (a)			3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil; former machine pit and sump
RISB-50	C-20, -21, -22	1-3 (f)	X	X (***)			X (*)			Std	X	X (*)		X (a)			3-Day	TBD	Further investigation near southwest side of C-22 where high levels of TCE were detected in soil gas and TCE was detected in soil; former machine pit and sump
RISB-29	C-23 & Annex	1-3 (f)	X	X (***)						Std	X	X		X (a)			3-Day	TBD	Further investigation in C-23 annex where high concentrations of TCE were detected in soil gas
RISB-30	C-23 & Annex	1-3 (f)	X	X (*)						Std	X	X		X (a)			3-Day	TBD	Dual purpose with Former C-29, TCE exceedances in the area
RISB-31	C-23 & Annex	1-3 (f)	X	X (***)		X (**)(d)	X (*)	X	X	Std	X	X		X (a)			3-Day	TBD	Dual purpose with Former C-29, TCE exceedances in the area
RISB-32	C-23 & Annex	1-3 (f)	X	X (*)						Std	X	X		X (a)			3-Day	TBD	Dual purpose with Former C-29, TCE exceedances in the area
RISB-33	C-23 & Annex	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std									Delineate vertical extent of contamination at LAI-14

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Table 16
Summary of Remedial Investigation Sampling and Analyses
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

		Soil									Groundwater							Soil Gas/ Sub Slab	Selection Rationale
Exploration ID	Investigation Area	# of Samples	VOCs	DRO / ORO	GRO	Metals	PCBs	TOC	Grain Size	TAT	VOCs	DRO / ORO	GRO	Dis. Metals	1,4-Dioxane	NA Params	TAT	VOCs	
RISB-34	C-23 & Annex	1-3 (f)	X	X (***)						Std									Delineation of petroleum hydrocarbon impacts, target floor joints or other points of potential release.
RISB-35	C-23 & Annex	1-3 (f)	X	X (***)						Std									Delineation of petroleum hydrocarbon impacts, target floor joints or other points of potential release.
RISB-36	C-23 & Annex	1-3 (f)	X	X (*)						Std	X	X		X (a)				TBD	Delineation of petroleum hydrocarbon impacts, target floor joints or other points of potential release.
RISB-37	C-23 & Annex	1-3 (f)	X	X (***)				X	X	Std	X	X (*)						TBD	Delineation of petroleum hydrocarbon impacts, target floor joints or other points of potential release.
RISB-38	C-23 & Annex	1-3 (f)	X	X (***)						Std	X							TBD	On edge of old equipment pad
RISB-39	C-23 & Annex	1-3 (f)	X	X (*)		X (**)(d)	X (*)			Std	X	X (*)						TBD	Further investigation in area of sump (LAI-28) where vinyl chloride was detected in soil gas
RISB-40	C-23 & Annex	1-3 (f)	X	X (*)		X (**)(d)	X (*)			Std	X	X (*)						TBD	Further investigation in area of sump (LAI-28) where vinyl chloride was detected in soil gas
RISB-51	C-23 & Annex	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X (*)						TBD	Further investigation in C-23 annex where high concentrations of TCE were detected in soil gas
RISB-52	C-23 & Annex	1-3 (f)	X	X (***)		X (**)(d)	X (*)			Std	X	X				X		TBD	West of aboveground storage tanks
RISB-53	C-23 & Annex	1-3 (f)	X	X (*)		X (**)(d)	X (*)			Std	X	X					3-Day	TBD	Delineation of petroleum hydrocarbon impacts, target floor joints or other points of potential release.
RISB-41	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)					Std	X	X	X	X (a)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-42	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)			X	X	Std	X	X	X	X (a)			3-Day	X	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-43	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)					Std	X	X	X	X (a)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-44	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)					Std	X	X	X	X (a)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-45	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)					Std	X	X	X	X (a)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-46	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)					Std	X	X	X	X (a)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-47	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)	X (**)(e)	X (*)			Std	X	X	X	X (b)			3-Day	TBD	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RISB-48	C-29/Fuel Farm	1-3 (f)	X	X (*)	X (*)	X (**)(e)	X (*)	X	X	Std	X	X	X	X (b)			3-Day	X	Delineate extent of previously identified contamination associated with former Building C-29 and the former fuel farm
RIDW-1	Deep Aquifer	3-5 (g)	X					X (j)	X (j)	Std	X (h)					X	Std		Evaluate aquifer conditions and delineate extent of contamination identified at DW1 and DW2
RIDW-2	Deep Aquifer	3-5 (g)	X					X (j)	X (j)	Std	X (h)					X	Std		Evaluate aquifer conditions and delineate extent of contamination identified at DW1 and DW2
RIDW-3	Deep Aquifer	3-5 (g)	X					X (j)	X (j)	Std	X (h)				X	X	Std		Evaluate aquifer conditions and delineate extent of contamination identified at DW1 and DW2

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Table 16
Summary of Remedial Investigation Sampling and Analysis
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Notes:

Boring Depth All borings will be advanced to approximately 5 ft below the fill-till interface unless contamination is observed at the bottom of the 5 ft penetration into the till.

Samples will be collected at a maximum interval of 5 ft. Borings have no maximum depth and will end in in soil with no observable contamination.

(a) Dissolved Metals: MTCA metals (arsenic, cadmium, chromium, lead, and mercury)

(b) Dissolved Metals: MTCA metals + Cr IV

(c) Natural Attenuation Parameters: nitrate, sulfate, methane, ethane, ethene, total organic carbon, and ferrous iron (kit)

(d) Total Metals: MTCA metals

(e) Total Metals: MTCA metals + Cr VI

(f) **For shallow soil borings**, the depth of drilling and the soil interval from which soil samples are collected for laboratory analysis will depend on the depth below ground surface to the fill/till interface and on the condition of the soil sample based on field screening as detailed below:

Depth to fill/till interface 0-10 ft bgs

- Drill to depth of fill/till interface plus 5 ft
- If field-observed contamination is present, collect one sample at depth with greatest field-observed contamination and at bottom of boring
- If no field-observed contamination is present, collect one sample at fill/till interface
- If field-observed contamination is present at depth of fill/till interface plus 5 ft, continue drilling until contamination is no longer observed and collect sample of presumed clean interval.

Depth to fill/till interface 10-20 ft bgs

- Drill to depth of fill/till interface plus 5 ft
- If field-observed contamination is present between 0 ft-10 ft, collect sample in this interval with greatest field-observed contamination; if no field-observed contamination is present, do not sample this inter
- If field-observed contamination is present between 10 ft and depth of fill/till interface plus 5 ft, collect one sample at depth with greatest field-observed contamination and at bottom of boring.
- If no field observed contamination is present between 10 ft and depth of fill/till interface plus 5 ft, collect one sample at fill/till interface
- If field-observed contamination is present at depth of fill/till interface plus 5 ft, continue drilling until contamination is no longer observed and collect sample of presumed clean interval.

(g) **For deep soil borings** (for the installation of deep monitoring wells), the depth of drilling will be approximately 150 ft BGS. Soil will be collected for laboratory analysis as detailed below:

- One sample will be collected from each of the following three depth intervals: 25 ft-50 ft, 50 ft-75 ft, and 75 ft-100 ft. The actual sample will be collected from the portion of the interval that yields the great
- One sample will be collected from the 100 ft-125 ft and 125 ft-150 ft depth intervals only if field-observed contamination is present. The actual sample will be collected from the portion of the interval that y

(h) grab sample and well sample to be analyzed

(i) If shallow groundwater is encountered, install monitoring well. No groundwater grab samples will be collected.

(j) Collect two samples per boring, one sample from the glacial till unit at approximately 50 ft bgs and one from the saturated zone.

(*) Collect sample only if field screening indicates hydrocarbon contamination, for soil collect sample from most contaminated zone.

(**) If evidence of petroleum contamination is observed, collect sample for metals at the impacted interval; if no evidence of contamination is observed, collect sample at the shallower of 2.5 ft bgs or fill/till interface

(***) If no evidence of contamination is observed then collect one sample at 2.5 ft or at fill/till interface, whichever is shallower; if evidence of contamination is observed, see footnote (f).

Std Standard Turnaround-Time

TBD If no groundwater encountered, collect soil gas and analyze for 1,1,1-TCA, 1,1-DCA, PCE, TCE, VC, and benzene

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Table 16
Summary of Remedial Investigation Sampling and Analysis
Remedial Investigation Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

rval.

est level of field-observed contamination. If no field-observed contamination is present, a sample will be collected from the approximate midpoint of the interval (e.g., 37-38 ft for the 25 ft-50 ft depth interval).
ields the greatest level of field-observed contamination.

APPENDIX A

Sampling and Analysis Plan

Draft
Sampling and Analysis Plan
Remedial Investigation/Feasibility Study Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

September 19, 2018

Prepared for

Snohomish County Public Works
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Sampling and Analysis Plan
Remedial Investigation/Feasibility Study Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

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LIST OF ABBREVIATIONS AND ACRONYMS

bgs	below ground surface
Ecology	Washington State Department of Ecology
EDB	ethlyene dibromide
EPA	US Environmental Protection Agency
ft	feet
IDW	investigation-derived waste
LAI	Landau Associates, Inc.
mL	milliliter
mL/min	milliters per minute
PCB	polychlorinated biphenyl
PID	photoionization detector
ppm	parts per million
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
RI	remedial investigation
SAP	sampling and analysis plan
TECT	TECT Aerospace
TPH	total petroleum hydrocarbons
VOC	volatile organic compound
USCS	Unified Soil Classification System
WAC	Washington Administrative Code
work plan	Remedial Investigation/Feasibility Study Work Plan

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1.0 INTRODUCTION

Landau Associates, Inc. (LAI) prepared this sampling and analysis plan (SAP), which describes the procedures for conducting field activities during the remedial investigation (RI) at the TECT Aerospace (TECT) property (Site), located in Everett, Washington (Figure A-1). This SAP is an appendix to the TECT Aerospace remedial investigation work plan (work plan). The primary objective of this SAP is to provide sampling and analysis procedures and methodologies consistent with accepted procedures such that the data collected will be adequate for use in characterizing environmental conditions at the Site. This SAP was prepared in accordance with the requirements of Washington Administrative Code (WAC) 173-340-820.

This SAP addresses RI field work, during which samples of soil, groundwater, and soil gas will be collected at the Site. The anticipated number of samples and analyses for each medium are summarized in Table 16 of the RI work plan. Table 16 describes the anticipated sampling activities for each of the following investigation areas:

- Building C-19
- Building C-20, C-21, C-22 Complex
- Building C-23 and C-23 Annex
- Former Building C-29 / Former East Fuel Farm
- Deep Aquifer.

The following sections describe the field procedures to be employed for the planned sampling activities.

2.0 DRILLING AND SAMPLING

Soil and groundwater grab samples will be collected from borings drilled using either a direct-push drilling, rotosonic drill, or hollow-stem auger drill rig. Shallow borings will be extended to the maximum depth of field-observed contamination or, if field-observed contamination is not present, to 5 ft below the fill-till interface. Borings associated with the deep aquifer investigation will extend to approximately 150 feet (ft) below ground surface (bgs).

The borings will be completed by a driller licensed in the State of Washington and will be monitored by an LAI-environmental professional. Soil will be described and classified in accordance with the Unified Soil Classification System (USCS). Prior to initiation of drilling or any other intrusive subsurface activity, the locations of each proposed exploration will be checked in the field by reviewing information from Snohomish County to locate aboveground and/or underground utilities or physical obstructions that would prevent drilling at the proposed location. In addition, a public utility locate service will be contacted to locate underground utilities at the perimeter of the Site and a private utility locate service will be retained to survey the exploration locations and mark underground utilities. The final location for each borehole will be selected based on the findings of the field check and the utility locating and marking. Before and between drilling of each boring and at the completion of the project, downhole drilling equipment will be cleaned using a high-pressure hot water or steam washer, as described in Section 7.3.

During drilling, continuous soil samples will be collected at each soil boring location to classify soil lithology in accordance with the USCS. For soil borings advanced using a hollow-stem auger rig, the soil samples will be obtained using a 3-inch-diameter, 1.5-ft-long, split-spoon sampler. For soil borings advanced using a direct-push drill rig, soil samples will be collected using a closed-piston sampling device with a 48-inch-long, 1.5-inch-diameter core sampler. For soil borings advanced with a rotosonic drill rig, soil samples will be collected in 5-to 20-ft core barrels; core barrel diameters range from 3 to 8 inches depending on the soil clast size and drill rig limitations.

A record of the soil and groundwater conditions observed during drilling will be recorded on a Log of Exploration form. The boring log will also show soil types; evidence of contamination based on field screening; and other pertinent information.

2.1 Field Screening

Soil, groundwater, and soil gas will be field-screened for evidence of environmental impact. Field-screening techniques may include visually inspecting the soil or groundwater for staining, discoloration, and other evidence of environmental impact; monitoring soil gas for volatile organic compounds (VOCs) using a portable photoionization detector (PID); observation of odors; and sheen testing. Field screening will be conducted at all exploration locations on all media. VOC monitoring for soil will be conducted using headspace analysis and will be performed by first measuring VOC levels along the length of freshly exposed soil in recovered soil cores (e.g., split spoon sampler, core sampler, or core barrel) using the PID. If VOC readings above background levels are observed, a small amount of soil from that portion of the soil core yielding the VOCs will be placed in a Ziploc® bag. The bag will then be sealed, the contents broken up, and the bag allowed to equilibrate for 2 to 5 minutes. The tubing from a PID will then be inserted into the Ziploc bag, the bag will be resealed around the tube, and the highest reading measured by the PID will be recorded. A PID will also be used to screen soil gas for VOCs during purging prior to sample collection. Sheen testing will be performed by agitating a small volume of soil in a stainless steel bowl with clean water to see if a sheen is generated. Groundwater samples will be observed for evidence of sheen, odor, and discoloration.

All field-screening results will be recorded and entered in the comments section of the soil boring logs. Additionally, any PID readings over 5 parts per million (ppm) will be noted on the chain-of-custody form to communicate the potential for contamination to the laboratory.

2.2 Soil Sampling

This section discusses soil sampling methodology. Please refer to Section 6.0 of the work plan for a discussion of soil sampling locations, sampling intervals, and analytical methods. Sample containers, labeling, and handling methods are discussed below.

Soil samples to be tested for non-volatile parameters (i.e., metals, polychlorinated biphenyls [PCBs], and diesel- and oil-range total petroleum hydrocarbons [TPH]) will be collected from the identified soil sampling intervals using the following methods:

- Scrape the outside of the soil core to expose a fresh sampling surface using a clean, decontaminated stainless steel spoon.
- Homogenize the soil in a decontaminated stainless steel bowl using the stainless steel spoon.
- Transfer the homogenized soil into the appropriate laboratory-supplied sample container.

Note that collection of soil samples for analysis for TPH at some locations is not planned unless field-screening results indicate the potential presence of VOCs.

Soil samples collected for analysis for volatile parameters (e.g., gasoline-range TPH and any VOCs including benzene, toluene, ethylbenzene, and xylenes) will be collected in accordance with US Environmental Protection Agency (EPA) Method 5035A. The EPA 5035A soil sampling method is intended to reduce volatilization and biodegradation of samples. The EPA 5035A procedure for soil sample collection is as follows:

- Collect soil cores from the split-spoon sampler using coring devices (i.e., EnCore® sampler, EasyDraw Syringe®, or a Terra Core™ sampling device). Each core will consist of approximately 5 grams of soil. Collect three discrete cores from each sampling location. One EasyDraw Syringe or Terra Core device will be used to collect the three discrete cores; however, if the EnCore samplers are used, then three sampling devices are required.

- Remove excess soil from the coring device. If an EasyDraw Syringe or Terra Core sampling device is used for sample collection, then place the “cored” soil directly into three preserved 40 milliliter (mL) vials with a stirbar. Vials will be preserved as indicated in Table A-1. If the EnCore sampler is used, then close the sampler for transport to the laboratory.
- Collect 2 ounces of soil and place in a laboratory-supplied jar for moisture content analysis and laboratory screening purposes. Fill the jar to minimize headspace.

Soil samples collected for laboratory analysis will be labeled using the following format:

> “RISB-location-(depth interval ‘)”
> For example, a soil sample collected between 4 and 5 ft bgs at RISB-6 would be RISB-6-(4-5’).

2.3 Groundwater Sampling

This section discusses groundwater grab sampling methodology. Please refer to Section 6.0 of the work plan for a discussion of groundwater grab sampling locations, sampling intervals, and analytical methods. Sample containers, labeling, and handling methods are discussed below.

2.3.1 Groundwater Grab Sampling

Groundwater grab samples will be collected for laboratory analysis from borings as described in Table 16 of the RI work plan. If direct-push drilling is used, the groundwater samples will be collected using a groundwater sampler consisting of a 4-ft-long, wire-wrapped, stainless steel screen (0.010-inch slot size) with a retractable protective steel sheath. The groundwater sampler will be advanced to the sample depth and the protective sheath will be retracted to expose the stainless steel screen to the formation. If rotosonic or hollow-stem auger drilling is used, temporary monitoring wells with PVC screens will be constructed in the boreholes and used for groundwater sample collection.

Low-flow purging will be conducted using a peristaltic pump for 10 minutes or until the purge water is clear. During purging, pH, conductivity, and temperature will be measured using a flow-through cell and recorded on a field sample collection form. Groundwater samples will be collected directly into the appropriate sample containers using disposable polyethylene tubing and the peristaltic pump. Samples will be chilled to 6°C immediately after collecting the sample. Groundwater for dissolved metals analyses will be collected last and field-filtered through a 0.45-micron, in-line disposable filter. A note will be made on the sample label, sample collection form, and chain-of-custody form to indicate that the sample has been field-filtered.

The groundwater grab samples collected from the soil borings will be labeled using the following format:

> “RISB-location-GW”
> For example, a groundwater grab sample collected at RISB-6 would be RISB-6-GW.

2.4 Soil Gas Sampling

Procedures for soil gas sampling follow the recommendations in the Washington State Department of Ecology (Ecology) draft guidance document on vapor intrusion evaluation (Ecology 2018)[1], and are detailed below.

2.4.1 Soil Gas Well Installation

2.4.1.1 Outside of a Building Footprint

Soil gas wells located outside of a building footprint will be installed by a licensed drilling contractor using direct-push or auger drilling techniques. Soil gas wells will consist of 0.25-inch-diameter Teflon® or Nylaflow® tubing with a 6-inch-long, stainless steel vapor implant. Vapor implants will be installed to a depth of 5 ft bgs or at the till-fill interface, whichever is shallower. If groundwater is encountered, the implant will be set at least 1 ft above the groundwater table. Vapor implants will not be set shallower than 2.5 ft bgs outside of the building footprint. Water levels will be confirmed at the time of field installation (in nearby wells and in groundwater grab sample borings) so that soil gas wells are constructed to the appropriate depth.

Silica sand will be placed around the screened interval to depths of at least 6 inches above and below the implant. Approximately 6 inches of dry granular bentonite will be installed above the filter pack. Borings will be sealed from the top of the dry granular bentonite to the ground surface with hydrated cement/bentonite grout. Soil gas wells will be equipped with dedicated valves, and completed with traffic-rated flush-mount monuments. All dedicated materials (tubing, probe screen, valve) will be delivered from the manufacturer pre-cleaned and sealed in plastic.

2.4.1.2 Sub-Slab

Sub-slab vapor sampling points (within a building slab) will be constructed by drilling a hole through the concrete floor slab, inserting a sample collection device, and sealing the hole around the sample collection device so that ambient air cannot enter the subsurface. A sample point may be constructed in one of two ways, either by inserting a Cox-Colvin Vapor Pin™ (Vapor Pin) of approximately 3 inches in length into the hole, or by placing a vapor implant into the hole and sealing the hole with hydrated bentonite. The sections below describe the installation process for both the Vapor Pin and the vapor implant. The installation sections are followed by a description of the sampling protocol, which is the same for both installation types.

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[1] Ecology. 2018. Review Draft: Guidance for Evaluating Soil Vapor Intrusion in Washington State: Investigation and Remedial Action. Publication No. 09-09-047. Washington State Department of Ecology. Revised April. https://fortress.wa.gov/ecy/publications/documents/0909047.pdf.

Sub-slab soil gas samples will be collected from just beneath or within a slab from a ⅝-inch- or 1-inch-diameter hole. The hole will be drilled with a handheld rotary hammer style drill.[2] Immediately following coring, field staff will insert a PID into the drilled hole to quickly check for VOCs, and will proceed with installing the sample point to minimize the introduction of soil gas into indoor air as described below:

- **Cox-Colvin Vapor Pin -** Vapor Pins are composed of a barbed, stainless steel sample point fitted with an inert, compressible, silicon sleeve. Each Vapor Pin will be installed using a hammer and specialized installation tool to drive the Vapor Pin into a ⅝-inch-diameter vertical hole within the slab. Driving the Vapor Pin into the hole compresses the sleeve, creating a seal between the sample point and slab surface. Typically, slabs are thicker than 3 inches, so the bottom of the Vapor Pin will rest within the slab, above underlying soil. After the Vapor Pin is installed, the end with a hose barb is exposed at the ground surface. A fitted cap will be attached to the barb to allow the sub-slab soil gas to equilibrate without exposure to ambient air. A flush-mounted installation will be used for locations where multiple sampling events are anticipated.
- **Vapor Implant -** Vapor implants are typically installed in a 1-inch-diameter vertical hole within the slab; larger holes may be used, but are not anticipated for this sampling event. Vapor implants comprise a porous sampling tip and sample tubing placed in the core hole. Teflon or Nylaflow tubing may be used as sample tubing. The porous sampling tip will extend 1 to 2 inches below the bottom of the slab. The void space around the sampling tip will be backfilled with drilling grade silica sand up to the bottom of the slab. Granular bentonite will be used to seal the annular space between the sample tubing and the slab within the hole. Two to 3 inches of dry bentonite will be placed on top of the silica sand. Tap water will be used to hydrate the top 1 inch of bentonite. A pipet or other small implement that can carefully control water flow will be used to administer the water and care will be taken to avoid getting water into the silica sand layer. Once the top layer of bentonite is hydrated, additional bentonite or expanding cement will be added and hydrated in 1- to 2-inch lifts until it is within ½ inch of the top of the slab. A flush-mounted installation will be used for locations where multiple sampling events are anticipated. The tubing will be secured with a valve and the core will be capped between sampling events.

2.4.2 Soil Gas Sample Collection

Sample collection activities at soil gas wells will occur after an equilibration period (at least 1 hour after installation of sample points) to allow for soil gas conditions to return to steady-state prior to sample collection. Soil gas sampling will not be performed during or immediately following a heavy rain event. Sampling will not be conducted within 48 hours following a rain event with more than 0.5 inches of rain in 24 hours.

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[2] Some coring debris will remain at the bottom of the boring; therefore, drilling should extend beneath the bottom of the slab by approximately 4 to 6 inches to expose the soil before installing the Vapor Pin or implant. Sub-slab conditions (such as Site-specific construction features) may require shallower drilling beneath the sub-slab, which will be evaluated at each sample location and/or in each building prior to installation of the sample collection device. A broom and dust pan or shop vacuum will be used to collect coring debris deposits on the ground surface; a shop vacuum will be used only to clear the hole prior to breakthrough of the floor slab or after installation of the sample collection device.

Following the equilibration period, a shut-in test, helium leak test, and purge will be completed prior to sample collection, as detailed below.

2.4.2.1 Shut-In Test

The purpose of a shut-in test is to check the air-tightness of all connections in the sample train prior to conducting the helium leak test. The shut-in test will be completed using the following steps:

1. After the equilibration period is complete, connect the entire sample train to the soil gas implant tubing. The sample train will consist of the Summa canister, flow regulator (if necessary), vacuum gauge assembly, valves, tubing, and syringe. Double-check the tightness of all connections.
2. Use the syringe to pull a vacuum on the sample train. When both the in-line and canister vacuum gauges indicate a vacuum of at least 15 inches of mercury, record the shut-in test starting vacuum pressure and time. Continue to watch for vacuum drops for 1 minute. Record the shut-in test ending vacuum and time. If the vacuum holds steady with no observable drop for 1 minute, this indicates that there are no leaks in the sample train between the sample tubing and the Summa canister.
3. If the vacuum pressure drops during the shut-in test period, a leak is present. Double-check the tightness of fittings, examine tubing and other equipment for defects or other possible leaks, and repeat the test. If the pressure continues to drop, replace the sample train pieces in the following order: tubing, valves, canister.

2.4.2.2 Helium Leak Test

The helium leak test is optional and may be excluded if potential subsurface interferences are present such as significant methane. The helium leak test procedure described below requires one Dielectric Helium Leak Detector MGD 2002 (or equivalent) hand-held meter. Select the appropriate height shroud to fully encapsulate the aboveground portion of the sample port. Place the shroud over the sample port and extend the sample tubing through the outlet at the top of the shroud. Hydrated bentonite or bentonite clay can be used around the base of the shroud if an uneven ground surface is present. Follow the steps below:

1. Connect the helium tank to the shroud through an inlet port and ensure that all connections are tight.
2. Turn on the helium detector and zero-out the instrument in ambient air to read a helium concentration of 0 ppm. Insert the meter probe inside the shroud.
3. Release helium into the shroud until the helium detector indicates that the air inside the shroud is approximately 10 percent helium. Record the highest concentration of helium in ppm.
4. The same syringe used in the shut-in test can also be used for the helium test. Using the syringe, purge the sample train of at least one volume of air and discharge to ambient air. Then draw approximately 400 milliliters of soil gas into the Tedlar® bag. Close the Tedlar bag and remove it from the sample train.

5. Remove the helium detector from the sample shroud. Zero-out the instrument to read a helium concentration of 0 ppm.
6. Insert the helium detector into the Tedlar bag to measure the helium concentration in the extracted soil gas. The concentration of helium in the Tedlar bag should be zero.
7. If helium is detected in the Tedlar bag sample, a leak may be present in the surface seal, allowing ambient air to enter the well. Double-check the surface seal, tightness of fittings, and other possibilities for leaks. Consider adding a water dam or other additional surface seal material. Repeat the leak test using a new Tedlar bag and syringe. If helium continues to be detected in the Tedlar bag, there is the potential that methane (or another gas) is present in the subsurface formation that is causing interference with the helium detection meter. To check for an interfering gas, remove the helium shroud, disconnect the sample train from the sample tubing coming out of the ground, and check for helium directly from the sample tubing. If helium is detected, there is likely an interfering gas present. Reconnect the sample train, reinstall the helium shroud, rerun the shut-in test, collect the sample, and add analysis for helium to the chain-of-custody form for this sample location. If helium is not detected in the formation gas, there may be a problem with the surface seal that allowed helium to get into the formation during the helium leak test. Assess the surface, look for damage or areas that could be sealed with hydrated bentonite, seal as appropriate, and rerun the helium leak test. If helium is still present, discuss the next steps with the project manager.

Following completion of the shut-in-test and helium leak test, the entire sample train has been tested for leaks.

2.4.2.3 Purge

1. Use the syringe to purge 3 volumes from the sample train (approximately 6 milliliters per ft of ¼-inch sample tubing). Repeat the process if the required purge volume is greater than the capacity of the syringe.
2. Record the purge volume.

2.4.2.4 Sample Collection

1. Open the Summa canister valve to begin collecting the sample. Record the time the canister was opened and the initial volume of the canister.
2. Once the vacuum gauge on the Summa canister reaches 5 inches of mercury, close the valve on the Summa canister. Ensure the Summa canister valve is closed prior to disconnecting it from the well to prevent accidental entrance of remnant low-level helium from the shroud into the Summa canister.

2.4.3 Soil Gas Sample Handling and Analysis

Samples will be delivered to the laboratory for analysis of the compounds listed in Table 16 of the work plan by EPA Method TO-15. In the event that a helium leak test cannot be completed as described above, the sample will be analyzed for helium by Method ASTM D-1946. Soil gas samples will be collected in 1-liter Summa canisters, provided by the analytical laboratory. Each Summa canister used for sample collection will be certified to contain less than the reporting limit for each of the target compounds.

Prior to sampling, each soil gas sample will be assigned a unique alphanumeric identifier as follows:

> Soil Gas
> “RISG-location-YYMMDD”
> For example, a soil gas sample collected at RISG-1 on October 5, 2018 would be RISG-1-181005.

Analytical methods, sample containers, holding times, and preservation requirements are provided in Table A-1.

3.0 MONITORING WELL GROUNDWATER SAMPLING

Procedures for installing and developing monitoring wells, and collecting groundwater samples from the monitoring wells are described below. Well construction details are provided as Figures A-1 (shallow wells) and A-2 (deep wells).

3.1 Installation and Construction of Monitoring Wells

Boreholes for the groundwater monitoring wells will be drilled using rotosonic or hollow-stem auger drilling equipment. Shallow monitoring wells will be drilled to the depth of the fill-till interface. Deep monitoring wells will be drilled to approximately 150 ft bgs. Monitoring wells will be constructed by a licensed drilling contractor in the State of Washington, in accordance with the Minimum Standards for Construction and Maintenance of Wells (Chapter 173-160 WAC). Oversight of drilling and well installation activities will be conducted by an LAI environmental professional familiar with environmental sampling and construction of resource protection wells.

The monitoring wells will be constructed with 2-inch-diameter, flush-threaded, Schedule 40 PVC pipe. Each shallow well will be constructed with 5-ft screens. Deep aquifer wells will have a 10-ft screen set to cover the portion of the aquifer between 5 and 15 ft below the top of the deep aquifer water table.

The screen at each well will be constructed with a 0.020-inch machine-slotted PVC casing. A filter pack material consisting of pre-washed, pre-sized number 10/20 silica sand or 2/12 Monterey Beach sand will be placed from the bottom of the well to between 1 and 2 ft above the top of the screen. Filter pack material will be placed slowly and carefully to avoid bridging of material.

A bentonite seal will be placed above the filter sand pack material to within about 3 ft of ground surface. In shallow wells, the seal can be 100 percent bentonite chips, while in the deep aquifer wells the seal will consist of 5 percent bentonite grout placed over approximately 5 ft of bentonite chips to prevent the grout from infiltrating into the filter pack. Concrete will be used to backfill the boring to the subgrade for placement of the protective cover. The wells will be completed with flush-mounted protective casings.

The well names and the identification numbers assigned by Ecology will be marked on the well identification tags supplied by Ecology. The tags will be attached to each well casing (inside the well monument) following well installation.

3.2 Well Development

The monitoring wells will be developed after construction to remove formation material from the well borehole and the filter pack prior to groundwater level measurement and sampling. Development will be achieved by repeatedly surging the well with a surge block and purging the well until the water runs clear, and at least five well casing volumes have been removed. During development, the purged groundwater will be monitored for turbidity.

The wells will be developed until the turbidity of the purged groundwater decreases to 5 nephelometric turbidity units, if practicable. If the well dewaters during the initial surging and purging effort, one final well casing volume will be removed after the well has fully recharged, if practicable. Well development activities will be recorded on a well development form.

3.3 Groundwater Sample Collection

Groundwater samples will be collected at least 24 hours after well development. Water levels will be measured prior to sample collection as described in Section 5.0. Groundwater samples will be collected at each monitoring well using low-flow sampling techniques and the following procedures:

- Immediately following removal of each well monument cover, the well head will be observed for damage, leakage, and staining. Additionally, immediately following removal of the well head cap, any odors will be documented and the condition of the well opening will be observed. Any damage, leakage, or staining to the well head or well opening will be documented.
- The depth to groundwater will be measured from the top of the casing prior to extraction of water from the well, using the procedures described in Section 5.0.
- Prior to sampling, each well will be purged using a peristaltic pump (for shallow wells) or a non-dedicated bladder pump (for deep wells) that is attached to dedicated purge and sample collection tubing. Purging will begin with a low pumping rate. The pumping rate will be maintained at less than 1 liter per minute and with drawdown of less than 1 ft during purging. Purging will continue until specific conductance, pH, dissolved oxygen, and turbidity have stabilized, as described below.
- Field parameters, including pH, temperature, specific conductance, dissolved oxygen, oxidation reduction potential, and turbidity, will be continuously monitored during purging using a flow cell. Purging of the well will be considered to be complete when all field parameters become stable for three successive readings. The successive readings should be within +/– 0.1 pH units for pH, +/– 3 percent for conductivity, and +/– 10 percent for dissolved oxygen and turbidity.
- Purge data will be recorded on a groundwater sample collection form including purge volume; time of commencement and termination of purging; any observations regarding color, turbidity, or other factors that may have been important in evaluating sample quality; and field measurements of pH, specific conductance, temperature, dissolved oxygen, and turbidity.
- Following the stabilization of field parameters, replicate measurements will be recorded on the sample collection form. Then, the flow cell will be disconnected and groundwater samples will be collected. Sample data will be recorded on a groundwater sample collection form, including sample number and time collected, the observed physical characteristics of the sample (e.g., color, turbidity, odor, and sheen), and field parameters (pH, specific conductance, temperature, dissolved oxygen, and turbidity).
- Any problems or significant observations will be noted in the “comments” section of the groundwater sample collection form.

- Groundwater samples will be collected directly into the appropriate sample containers using the same pump used for purging. To prevent degassing during sampling for VOCs, a pumping rate will be maintained below about 100 milliliters per minute (mL/min). The VOC containers will be filled completely so that no head space remains. Samples will be chilled to 4°C immediately after collection. Clean gloves will be worn when collecting each sample.
- Groundwater for dissolved metals analyses will be collected last and field-filtered through a 0.45-micron, in-line disposable filter. Groundwater samples for dissolved metals analysis will be preserved, as specified in Table A-1. A note will be made on the sample label, sample collection form, and chain-of-custody form to indicate the sample has been field-filtered.

Groundwater samples collected from new shallow monitoring wells for laboratory analysis will be labeled using the following format:

<u>Shallow Groundwater Wells</u>
"RIGW-location-YYMMDD"
For example, a groundwater sample collected at RIGW-1 on October 5, 2018 would be RIGW-1-181005.

<u>Deep Groundwater Wells</u>
"RIDW-location-YYMMDD"
For example, a groundwater sample collected at RIDW-3 on October 5, 2018 would be RIDW-3-181005.

4.0 LABORATORY ANALYSIS FOR SOIL AND GROUNDWATER

Soil samples will be selectively analyzed for one or more of the following: VOCs; metals; diesel-, oil-, and gasoline-range TPH; PCBs; ethylene dibromide (EDB); total organic carbon; and grain size. Groundwater samples will be selectively analyzed for VOCs; dissolved metals; diesel-, oil-, and gasoline-range TPH; EDB; and natural attenuation parameters nitrate, sulfate, methane, ethane, ethene, total organic carbon, and ferrous iron. The dissolved metals analysis will vary and will consist of the Model Toxics Control Act metals (arsenic, cadmium, chromium, lead, and mercury) plus chromium III and VI. Specific analyses by sample location are provided in Table 16 of the RI work plan. The analytical methods for the above-named compounds are summarized in Table B-3 of the QAPP (Appendix B of the work plan).

5.0 GROUNDWATER FLOW MONITORING

To evaluate groundwater flow direction, depth to groundwater will be measured at monitoring wells during each sampling event and at least once during the wet season and once during the dry season. This section describes the monitoring well survey and water level measurement procedures needed to evaluate groundwater flow direction.

The location of each new monitoring well will be surveyed using GPS equipment to facilitate accurate placement of these features on project figures and drawings. GPS surveying will be conducted after the wells have been installed.

Monitoring well reference elevations will be surveyed by LAI or by a professional licensed surveyor to the nearest 0.01 ft for use in evaluating groundwater and lithologic unit elevations. Both the top of the monitoring well PVC casing elevation and ground surface elevation adjacent to the monitoring well will be measured. Top of casing elevations will be used to develop groundwater elevation contour maps.

Water level measurements will be collected at each of the monitoring wells before the sampling event commences. All water levels will be measured using an electronic water level indicator and will be recorded to the nearest 0.01 ft. Measurements will be taken from the top of the north side of the well casing.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Analytical samples collected during the RI will follow quality assurance/quality control (QA/QC) procedures and standards outlined in the QAPP (Appendix B of the work plan). Field QA/QC includes the collection of QC samples consisting of blind field duplicate samples, matrix spike and matrix spike duplicate samples, and trip blanks. The procedures for collection of the QC samples are provided in the QAPP (Appendix B of the work plan). Sample containers, preservatives, and holding times for each chemical analysis are provided in Table A-1.

7.0 EQUIPMENT DECONTAMINATION

The decontamination procedures described below are to be used by field personnel to clean drilling, sampling, and related field equipment. Deviation from these procedures must be documented in field records.

7.1 Water Level Indicator

The tape from the water level indicator will be rinsed with Alconox® soap and distilled water between each well measurement.

7.2 Sampling Equipment

All sampling equipment used (e.g., stainless steel bowls, stainless steel spoons, soil split-spoon samplers, etc.) will be cleaned using a three-step process, as follows:

1. Scrub surfaces of equipment that would be in contact with the sample with brushes using an Alconox and water solution.
2. Rinse and scrub equipment with clean tap water.
3. Rinse equipment a final time with de-ionized water to remove tap water impurities.

Decontamination of reusable sampling devices (i.e., non-dedicated bladder pumps, etc.) will occur between each sample collection and will follow the above steps. At least 5 gallons of each decontamination liquid will be pumped through non-dedicated pump systems that cannot be fully disassembled.

7.3 Heavy Equipment

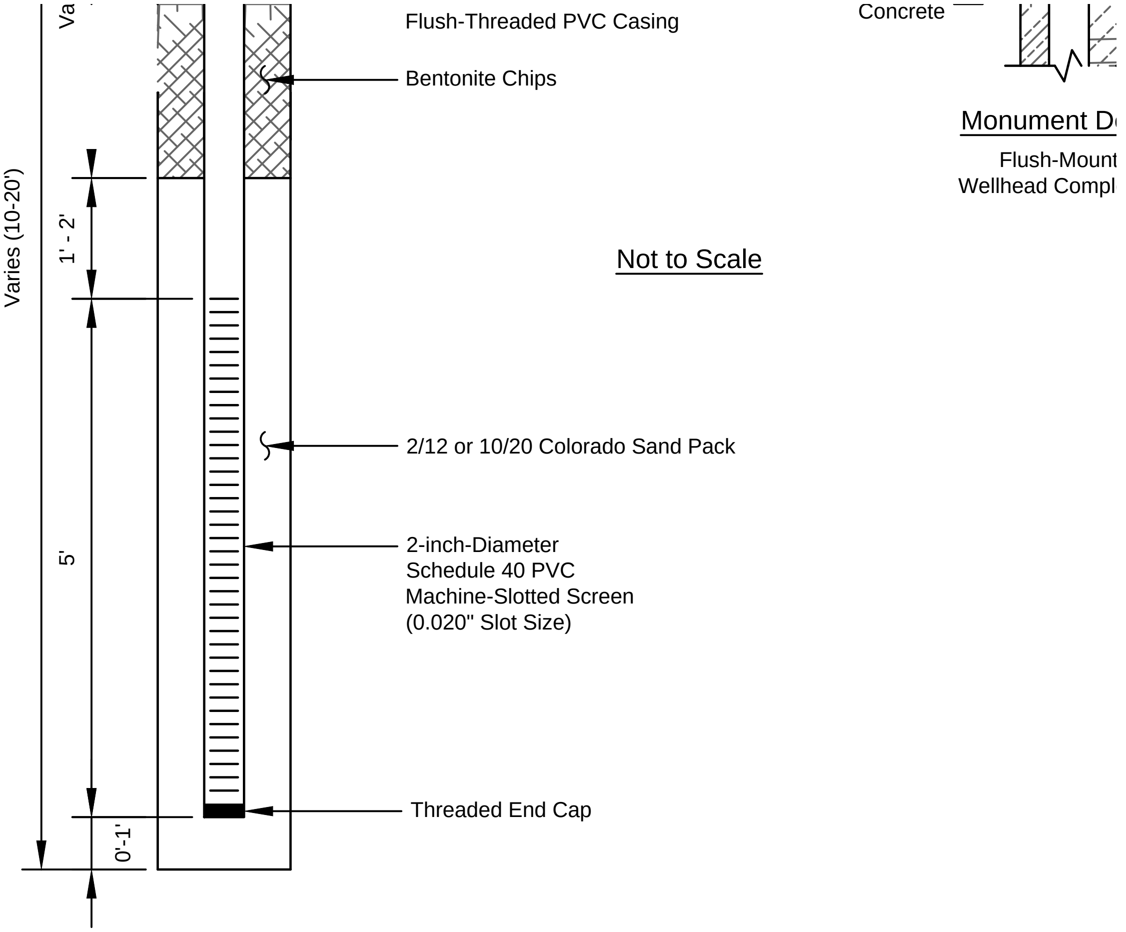
Heavy equipment (i.e., drilling equipment that is used downhole, or that contacts material and equipment going downhole) will be cleaned by a hot water, high-pressure wash before each use and at completion of the project. Potable tap water will be used as the cleaning agent.

8.0 RESIDUAL WASTE MANAGEMENT

Investigation-derived waste (IDW), including soil cuttings and water generated during drilling and sampling, and waste/wastewater generated during decontamination of sampling equipment or devices, will be collected and managed in containers provided by the driller. All waste will be characterized in accordance with applicable regulations based on the laboratory analytical results and historical knowledge. All IDW will be disposed of at facilities approved by Snohomish County and in accordance with applicable regulations.

9.0 USE OF THIS SAMPLING AND ANALYSIS PLAN

This Sampling and Analysis Plan has been prepared for the exclusive use of Snohomish County Public Works and applicable regulatory agencies for specific application to the former Paine Field TECT Aerospace Leasehold. No other party is entitled to rely on the information, conclusions, and recommendations included in this document without the express written consent of LAI. Further, the reuse of information, conclusions, and recommendations provided herein for extensions of the project or for any other project, without review and authorization by LAI, shall be at the user's sole risk. LAI warrants that within the limitations of scope, schedule, and budget, our services have been provided in a manner consistent with that level of care and skill ordinarily exercised by members of the profession currently practicing in the same locality under similar conditions as this project. We make no other warranty, either express or implied.



| G:\Projects\222\057\010\013\RI Work Plan\F0A-1ShallowMWDetail.dwg (A) "Figure A-1" 9/5/2018

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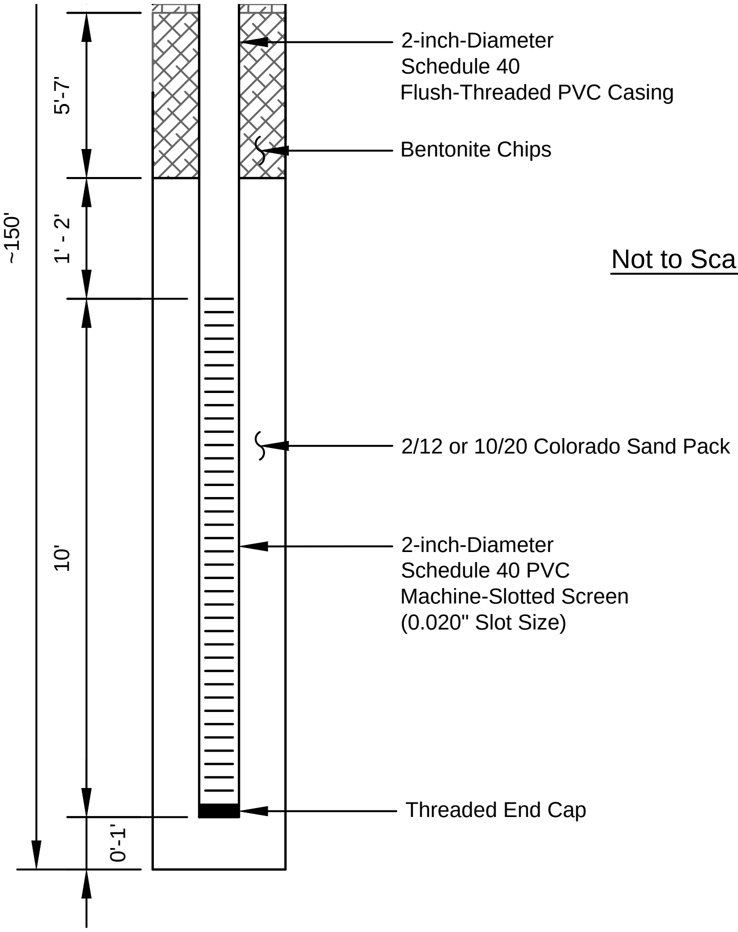
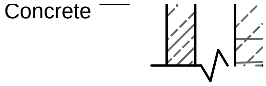
LANDAU
ASSOCIATES

TECT Aerospace
Everett, Washington

Shallow Monitoring Well
Construction Detail





Monument D

Flush-Mount
Wellhead Compl

Not to Scale

LANDAU ASSOCIATES

TECT Aerospace
Everett, Washington

Deep Monitoring Well Construction Detail

DRAFT

Table A-1
Sample Containers, Preservatives, and Holding Times
Sampling and Analysis Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Matrix	Method	Container	Preservative	Holding Time (a)	Laboratory Performing Analyses
Soil	Gasoline-range Petroleum Hydrocarbons by NWTPH-Gx	4 oz + 5035 methanol vial	<6^{o}C	14	ALS Global Everett
Groundwater	Gasoline-range Petroleum Hydrocarbons by NWTPH-Gx	2 x 40-mL glass	Add HCl to pH<2; <6^{o}C	14	ALS Global Everett
Soil	Diesel- and Oil-range Petroleum Hydrocarbons by NWTPH-Dx	4 oz	<6^{o}C	14 days/40 days	ALS Global Everett
Groundwater	Diesel- and Oil-range Petroleum Hydrocarbons by NWTPH-Dx	500-mL amber glass	<6^{o}C	7 days/40 days	ALS Global Everett
Soil	Total Metals by EPA 6020A (7471A for mercury)	4 oz amber glass	<6^{o}C (mercury only)	180 (mercury 28 days)	ALS Global Everett
Soil	Hexavalent Chromium by EPA 7196	4 oz polyethylene or glass	<6^{o}C	30 days to extraction then 24 hours for analysis	ALS Global Everett
Groundwater	Dissolved Metals by EPA Method 200.8 (245.1 for mercury)	500 mL plastic	If field filtered, HNO$_3$ to pH <2; <6^{o}C	180 (mercury 28 days)	ALS Global Everett
Groundwater	Hexavalent Chromium by EPA 7196	500 mL polyethylene or glass	<6^{o}C	24 hours	ALS Global Everett
Soil	VOCs by EPA Method 8260/8011	4 oz, + 5035 methanol vila and two stirbar vials	HCl to pH<2; <6^{o}C	14 days freeze stirbar vials within 48 hours	ALS Global Everett
Groundwater	VOCs by EPA Method 8260/8011	3 x 40-mL glass	HCl to pH<2; <6^{o}C	14 days (7 days pH >2)	ALS Global Everett
Groundwater	Nitrate by EPA Method 300.0	500 mL polyethylene	<6^{o}C	48 hours	ALS Global Everett

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Table A-1
Sample Containers, Preservatives, and Holding Times
Sampling and Analysis Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Matrix	Method	Container	Preservative	Holding Time (a)	Laboratory Performing Analyses
Groundwater	Sulfate by EPA Method 300.0	500 mL polyethylene	<6ºC	28	ALS Global Everett
Soil	Total Organic Carbon by ASTM D4129-05 Modified	4 oz amber glass	<6ºC	28	ALS Global Kelso
Groundwater	Total Organic Carbon by Method SM 5310C	500 mL amber glass	H_2SO_4 to pH <2; <6ºC	28	ALS Global Kelso
Groundwater	Natural Attenuation Parameters (methane, ethane, ethene, acetylene) by Method RSK-175	3 x 40-mL amber glass	HCL to pH <2; <6ºC	14	ALS Global Everett
Soil	PCBs by EPA Method 8082A	4 oz polyethylene or glass	<6ºC	365 days/40 days	ALS Global Everett

Acronyms/Abbreviations:

ºC = degrees Celsius
EPA = US Environmental Protection Agency
g = gram
H_3PO_4 = Phosphoric acid
HCL = Hydrochloric acid
HNO_3 = nitric acid
L = liter
mL = milliliter
oz = ounces
cPAH = carcinogenic polycyclic aromatic hydrocarbor
PCB = polychlorinated biphenyl
SIM = selected ion monitoring
SVOC = semivolatile organic compound
VOC = volatile organic compound

Notes:

(a) Time from sample collection to extraction/Time from sample extraction to analysis

APPENDIX B

Quality Assurance Project Plan

Draft
Quality Assurance Project Plan
Remedial Investigation/Feasibility Study Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

September 19, 2018

Prepared for

Snohomish County Public Works
3000 Rockefeller Avenue
Everett, Washington

Landau Associates

130 2nd Avenue South
Edmonds, WA 98020
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Quality Assurance Project Plan
Remedial Investigation/Feasibility Study Work Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

This document was prepared by, or under the direct supervision of, the technical professionals noted below.

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Data Specialist

Document reviewed by: ________________________________ Danille Jorgensen
Quality Assurance Manager

Date:	September 19, 2018
Project No.:	0222057.010
File path:	P:/222/057/R/RI-FS Work Plan/QAPP
Project Coordinator:	Christopher C. Young

LANDAU ASSOCIATES

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ATTACHMENTS

LIST OF ABBREVIATIONS AND ACRONYMS

ANSI	American National Standards Institute
ASQC	American Society of Quality Control
ATS	Aviation Technical Services
CLP	Contract Laboratory Program
COC	chain of custody
DQI	data quality indicator
DQO	data quality objective
EDD	electronic data deliverable
EPA	US Environmental Protection Agency
EQuIS	Environmental Quality Information Systems
eV	electron volt
FS	feasibility study
HASP	health and safety plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
LAI	Landau Associates, Inc.
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDL	method detection limit
MQO	measurement quality objective
MS	matrix spike
MSD	matrix spike duplicate
PCB	polychlorinated biphenyl
PID	photoionization detector
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RI	remedial investigation
RL	reporting limit
RPD	relative percent difference
SAP	sampling and analysis plan
Site	TECT Aerospace Leasehold
SL	screening level
SOPs	Standard Operating Procedures
TECT	TECT Aerospace
TPH	total petroleum hydrocarbons
VOC	volatile organic compound
WAC	Washington Administrative Code

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1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) establishes the quality assurance (QA) objectives for the remedial investigation/feasibility study (RI/FS) being conducted at the TECT Aerospace (TECT) Leasehold (Site) located in the southeastern portion of Sector 5 at Paine Field in Everett, Washington (Figures 1 and 2). This plan presents the quality control (QC) procedures developed to meet project QA objectives.

1.1 Distribution List

The following list identifies those individuals to receive an electronic copy of the approved QAPP, as well as any subsequent revised versions of the documentation:

- Andrew Rardin – Project Manager for Snohomish County Public Works (andrew.rardin@co.snohomish.wa.us)
- Gerald Ninteman – Consultant (Landau Associates, Inc. [LAI]) Project Manager (jninteman@landauinc.com)
- Danille Jorgensen – Consultant (LAI) Quality Assurance Officer (djorgensen@landauinc.com)
- Rick Bagan – Project Coordinator for ALS Global Laboratory Everett (rick.bagan@alsglobal.com)
- Sue Anderson – Project Coordinator for ALS Global Laboratory Simi Valley (sue.anderson@alsglobal.com).

1.2 Project Organization

Ongoing Site RI/FS activities will be implemented by Snohomish County Public Works; Andrew Rardin is the project manager. LAI is responsible for preparing documents associated with the planned RI/FS activities, implementing the activities, and reporting the RI/FS results to Snohomish County Public Works. Gerald Ninteman is the LAI RI/FS project manager and will communicate directly with Andrew Rardin, as necessary, during the course of RI/FS activities. Mr. Ninteman will be responsible for implementing and executing the technical, QA, and administrative aspects of the RI/FS and will manage LAI staff working on this project. Danille Jorgensen, the designated LAI Quality Assurance officer, is responsible for the overall management of the project-specific QA and QC requirements, including field and laboratory QC. LAI's staff managing field operations will report field progress and problems to Mr. Ninteman on a daily basis and will be responsible for managing subcontractors, as necessary, that support RI activities at the Site. Christine Kimmel is the designated Health and Safety Manager for field activities.

Specific QA responsibilities for this project are listed in Table 1. The QA manager will be responsible for QA oversight during investigation activities including sampling events, analytical laboratory coordination, and direct implementation of this QAPP. The QA manager will be responsible for overseeing data validation and for confirming that the QA objectives of the project are met.

2.0 PROJECT BACKGROUND / DESCRIPTION

The Site is approximately 10 acres in size and is located in the southeastern portion of the Snohomish County Airport. The Site is generally bounded by Runway 34-16 and associated taxiways on the east, a paved surface parking lot followed by Paine Field Road to the south, 109th Street and 30th Avenue South to the west, and the Aviation Technical Services (ATS) Hangar 1 lease area to the north. The former East Fuel Farm is located within the boundary of the ATS lease area.

The Site is currently zoned for light industrial use (Snohomish County; accessed July 17, 2018). Currently, the Site land is leased by Snohomish County to tenants for aerospace manufacturing and other light industrial operations.

Various environmental investigations and remedial actions have been conducted at the Site to characterize and evaluate the chemical quality and physical condition of soil, groundwater, and soil gas, or to address documented releases. Descriptions of previous activities are provided in the text of the RI/FS work plan.

2.1 Project Goals and Objectives

This QAPP has been prepared to cover work related to the RI/FS. The specifics of the RI/FS investigation are presented in the RI/FS work plan published concurrently with this QAPP.

The purpose of this QAPP is to provide specific QA and QC procedures that will be used to support the evaluation and interpretation of data determined to be of acceptable quality and completeness. This QAPP has been prepared based on the requirements outlined in the US Environmental Protection Agency's (EPA's) Guidance for Quality Assurance Project Plans (EPA 2002) and the EPA's Requirements for Quality Assurance Project Plans (EPA 2001).

To the extent possible, the procedures included in this QAPP have been standardized to support effective evaluation of data resulting from sampling of the various media that has the potential to be evaluated at the Site. In the event that additional investigation activities are performed following the publication of this QAPP (i.e., additional activities not addressed in the RI/FS work plan), work plan addenda will be prepared to document data quality objectives and sampling, and will include any revisions to screening levels (SLs), reporting limits (RLs), sampling procedures, and laboratories, as needed.

ALS Global Laboratories in Everett, Washington and Simi Valley, California are the laboratories to be used for planned RI/FS activities. Analytical testing will be in accordance with the methodologies established by the EPA (EPA 1974, 1999) and Standard Methods for the Examination of Water and Wastewater, 20th edition (APHA 1998). The EPA compendium of test methods (SW-846) provides for the analytical procedures to be used as well as the specific application of those procedures. Laboratory standard operating procedures (SOPs) are provided in Attachment 1.

3.0 QUALITY ASSURANCE OBJECTIVES

This section presents the QA and QC objectives and processes including data quality objectives (DQOs), data quality indicators (DQIs), measurement quality objectives (MQOs), and QC procedures for field and laboratory work.

3.1 Data Quality Objectives

DQOs specify the environmental decisions that the data will support and the corresponding level of data quality required to ensure decisions are based on sound scientific data. The DQOs for this project are determined by the area of investigation:

- Building C-19
    - Obtain data that are representative of Site conditions
    - Characterize concentrations of contaminants of concern in soil, groundwater, and soil gas, which include volatile organic compounds (VOCs); metals; polychlorinated biphenyls (PCBs); 1,4-dioxane; and/or diesel- and oil-range total petroleum hydrocarbons (TPH)
    - Obtain data that are comparable to applicable screening criteria
- Former Building C-29 and East Fuel Farm
    - Obtain data that are representative of Site conditions
    - Characterize concentrations of contaminants of concern in soil, groundwater, and soil gas, which include VOCs, metals (including hexavalent chromium), PCBs, and/or diesel-, oil-, and gasoline-range TPH
    - Analyses will also include characterization of natural attenuation parameters (nitrate, sulfate, methane, ethane, ethene, total organic carbon, and ferrous iron)
    - Obtain data that are comparable to applicable screening criteria
- Building C-23
    - Obtain data that are representative of Site conditions
    - Characterize concentrations of contaminants of concern in soil and groundwater, which include VOCs, metals, PCBs, and/or diesel- and oil-range TPH
    - Analyses will also include characterization of natural attenuation parameters (nitrate, sulfate, methane, ethane, ethene, total organic carbon, and ferrous iron)
    - Obtain data that are comparable to applicable screening criteria
- Building Complex C-20, C-21, and C-22
    - Obtain data that are representative of Site conditions
    - Characterize concentrations of contaminants of concern in soil and groundwater, which include VOCs, metals, PCBs, and/or diesel- and oil-range TPH
    - Obtain data that are comparable to applicable screening criteria

- Deep Aquifer Investigation
  - Obtain data that are representative of Site conditions
  - Characterize concentrations of contaminants of concern in deep soil and groundwater, which includes VOCs and 1,4-dioxane
  - Analyses will also include characterization of natural attenuation parameters (nitrate, sulfate, methane, ethane, ethene, total organic carbon, and ferrous iron)
  - Obtain data that are comparable to applicable screening criteria

3.2 Data Quality Indicators

DQIs are used to establish DQOs and are discussed in detail below. A summary of DQIs and their associated MQOs are presented by sample matrix in Table 2.

3.2.1 Precision

Precision is a measure of variability in the results of replicate measurements due to random error (Ecology 2004). Precision is best expressed in terms of the standard deviation or relative percent difference (RPD). QC sample types that can be used to evaluate precision include field and laboratory duplicates, matrix spike duplicates (MSDs), and laboratory control sample duplicates (LCSDs). The precision of duplicate measurements will be expressed as an RPD, which is calculated by dividing the absolute value of the difference of the two measurements by the average of the two measurements, and expressing it as a percentage. The formula for RPD calculation is shown below:

$$\mathrm{RPD} = \left[\frac{|\mathrm{D1} - \mathrm{D2}|}{[(\mathrm{D1} + \mathrm{D2}) \div 2]}\right] \times 100\%$$

Where:

D1 = first measurement value

D2 = second measurement value (duplicate).

3.2.2 Accuracy

Accuracy is a combination of precision and bias (described in Section 3.2.7), in that it represents the degree to which a measured value represents the known value (Ecology 2004). Accuracy is expressed as the percent recovery of spiked samples (matrix spike [MS], laboratory control sample [LCS], and surrogate spike). The general formula used to calculate percent recovery is shown below (for MS/MSD percent recovery, the result from the unspiked sample is taken into account in the formula):

$$\%\mathrm{R} = \left[\frac{\mathrm{SSR}}{\mathrm{C_s}}\right] \times 100\%$$

Where:

%R = percent recovery

SSR = spiked sample result

C_S = concentration of the spike added.

3.2.3 Representativeness

Representativeness is an indicator of how accurately a result reflects the desired characteristic(s) of a defined population, accounting for both temporal and spatial variability (Ecology 2004). Representativeness qualitatively describes how well the analytical data characterize an area of concern. Representativeness is largely determined by the sampling design; analytical parameters for use in its evaluation include method-specified holding times and preservation requirements, and matrix heterogeneity. The sampling design for this project is discussed in the RI/FS work plan.

3.2.4 Comparability

Comparability is the “degree of confidence with which one data set can be compared to another” (Ecology 2004). QC procedures and MQOs, as stated in this QAPP, will provide for measurements that are consistent and representative of the media and conditions measured.

3.2.5 Completeness

Completeness is a measure of “the amount of valid data obtained from a measurement system compared to the amount that could be expected to be obtained under normal conditions”(EPA 2009). Field completeness is calculated as the number of actual samples collected divided by the number of planned samples. Analytical completeness is calculated as the number of valid data points divided by the total number of data points requested. Data points are considered invalid if they are rejected during data validation. The data validation approach for this project is provided in Section 6.0. The requirements for field sampling and analytical completeness are 90 percent each.

3.2.6 Sensitivity

Sensitivity is the capability of a method or an instrument to discern the difference between very small amounts of a substance. For the purposes of this project, sensitivity is the lowest concentration that can be accurately detected by the analytical method. The analytical method will be considered sufficiently sensitive if the RLs are below the specific SLs for the area under investigation. In some instances, RLs are greater than SLs due to limitations of commonly used analytical technology. Proposed method and target RLs are presented in Tables 3 and 4. As necessary to meet project-specified SLs, sample results will be reported to the method detection limit (MDL). Results that are detected at concentrations between the MDL and the RL will be J-qualified as estimated.

3.2.7 Bias

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Bias of the laboratory results will be evaluated based on analysis of reference materials, method blanks, and MS samples, as described in Section 6.5.

4.0 SPECIAL TRAINING / CERTIFICATION

Personnel performing onsite investigation tasks will have completed formal 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) health and safety training, in compliance with 29 Code of Federal Regulations 1910.120 and Chapter 296 of the Washington Administrative Code (WAC). Certificates of successful completion of training, which will be maintained in personnel health and safety files, will verify on-the-job training for those tasks staff are assigned to perform. At least one member of each field team and the designated site safety officer will be trained in cardiopulmonary resuscitation and first aid.

Borings will be completed and monitoring wells will be constructed by a licensed drilling contractor in the state of Washington, following Washington State well standards. Oversight of drilling and well installation activities will be performed by an environmental professional familiar with environmental sampling and construction of resource protection wells.

As indicated in Section 2.1, ALS Global Laboratories in Everett, Washington and Simi Valley, California are the laboratories to be used for planned RI/FS activities. These laboratories are not in the Contract Laboratory Program (CLP), but are accredited through the Washington State Department of Ecology for the applicable methods and target analytes listed in this QAPP. Laboratories shall maintain current applicable state certification or US Department of Defense Environmental Laboratory Accreditation Program certification for the methods and target analytes listed in this QAPP while performing analyses for the project. Laboratories used for this project have a documented QA program that complies with standards promulgated by the American National Standards Institute/American Society of Quality Control (ANSI/ASQC 1994); ANSI's Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (Johnson 1994); and the EPA's Requirements for Quality Assurance Project Plans (EPA 2001).

Excavation, trenching, and shoring (WAC 296-155-Part N) activities or work in confined spaces (WAC 296-62-Part M) are not anticipated in this scope of work; therefore, this QAPP does not address training in physical worker safety issues that may be associated with excavation or confined spaces.

5.0 DOCUMENTS AND RECORDS

This section describes the management requirements for production, distribution, and storage of documents and records associated with planned activities at the Site.

5.1 Document Distribution

Prior to beginning field activities, field staff will receive and have an opportunity to review project-related documents pertinent to the field activities, including work plans, sampling and analysis plans (SAPs), and health and safety plans (HASPs), as appropriate to the planned activities. Project managers/coordinators will meet with field staff prior to field activities to review the relevant plans accordingly. The HASP will be reviewed in the field on the first day of activities, with each field person documenting their attendance to the HASP review on a sign-in sheet. The HASP will be reviewed again every few days or when a new field person begins working on field activities. The SAP, HASP, and work plans for each phase of the project will be finalized prior to commencement of field activities, and only the finalized versions will be distributed to field staff. Changes to procedures and plans after finalization will be documented as addenda and distributed along with the original finalized versions.

5.2 Field Documentation

Field equipment will have reference and related manuals stored in with the equipment. In addition, equipment that requires calibration will be accompanied by a calibration logbook. Field staff will record the calibration process in the logbook every time a calibration is performed.

A complete record of field activities will be maintained for the duration of the field phase of the work. Documentation will include the following:

- Daily recordkeeping by field personnel of field activities
- Recordkeeping of samples collected for analysis (field sampling forms)
- Use of sample labels and tracking forms for samples collected for analysis.

The field logs will provide a description of sampling activities completed, sampling personnel, daily weather conditions, and a record of modifications to the procedures and plans identified in the work plan or related documentation. The field logs are intended to provide sufficient data and observations to enable project staff to reconstruct events that occurred during the sampling period.

Field logs will be supplemented by sample collection forms, boring logs, and groundwater well logs completed by field staff, as applicable. The information that will be recorded in these forms is specified in the RI/FS SAP.

Additional records associated with drilling will include driller’s daily reports and well-related documentation when a well is installed.

Sample possession and handling will also be documented with chain-of-custody (COC) forms so that it is traceable from the time of sample processing in the field, to delivery to the laboratory, and to the ultimate data analysis. Sample handling and COC procedures are described in Section 6.3.

The following example field forms are provided in Attachment 2:

- Chain-of-Custody
- Field Report
- Groundwater Low-Flow Sample Collection Form
- Log of Exploration
- Log of Test Pit
- Soil/Sediment Sample Collection Form
- Survey Field Notes Form
- As-Built Well Completion Form
- Well Development Record Form.

5.3 Analytical Data Records

Laboratory analytical data reports will be provided in electronic format by the laboratory. These reports will be included as appendices in documents where data are reported, and will be kept along with all other documents in the project files. Data will be provided in a Level II laboratory report format. Data package elements are listed in Section 6.7.

5.4 Storage

Documents and records associated with the project (i.e., final documents, billing and invoice records) and the documents described in Sections 5.2 and 5.3 will be stored in electronic form in project files on LAI’s servers for the duration of the project.

6.0 DATA GENERATION AND ACQUISITION

This section provides an overview of the data collecting and handling processes that will ensure data quality that meets project standards. More details about these processes are included in the RI/FS SAP.

6.1 Sampling Process Design

A sampling design that achieves the DQOs described in Section 3.0 has been prepared and is detailed in the RI/FS SAP.

6.2 Sampling Methods and Containers

Samples will be collected using methods that are standard in environmental remediation. A detailed description of the sampling methods for each medium is provided in the RI/FS SAP. Methods for sampling, decontamination, and well installation are provided as SOPs in the RI/FS work plan, published concurrently with this QAPP.

Sampling containers will be provided by the laboratory. Extra containers will be requested to ensure that clean containers are available to replace any broken or misused containers during sampling events. The laboratory will provide kits (e.g., plunger for EPA Method 5035 soil sampling) to collect samples for analyses that require special methods to fill the sample container.

6.3 Sample Handling and Custody

Soil and water samples submitted to the analytical laboratories will be collected in the appropriate sample containers and preserved as specified in Table 5. The storage temperatures and maximum holding times for physical/chemical analyses are also provided in Table 5.

The transportation and handling of samples will be accomplished in a manner that not only protects the integrity of the sample, but also prevents any detrimental effects due to release of samples. Samples will be logged on a COC form (Attachment 2) and will be kept in coolers on ice until delivery to the analytical laboratory. The COC will accompany each shipment of samples to the laboratory. A sample is in custody if at least one of the following is true:

- It is in someone’s physical possession
- It is in someone’s view
- It is secured in a locked container or otherwise sealed so that tampering will be evident
- It is kept in a secured area, restricted to authorized personnel only.

Sample control and COC protocols in the field and during transport to the laboratory will be conducted in general conformance with the procedures described below:

- As few persons as possible will handle samples.

- Sample bottles will be obtained new or pre-cleaned from the laboratory performing the analyses.
- The sample collector will be personally responsible for the completion of the COC record and the care and custody of samples collected until they are transferred to another person or dispatched properly under COC rules.
- The onsite team leader will oversee implementation of the field custody procedures during the field work and, in the event of non-compliance, will determine if corrective action is required.
- The coolers in which the samples are shipped will be accompanied by the COC record identifying their contents. The original record and laboratory copy will accompany the shipment (sealed inside the shipping container). The other copy will be distributed as appropriate to LAI's QA officer or designee. The QA officer for this project is Danille Jorgensen.
- Shipping containers will be sealed with custody seals for shipment to the laboratory. The method of shipment, name of courier, and other pertinent information will be entered in the "remarks" section of the COC record.
- If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a bill of lading will be used. Freight bills, postal services receipts, and bills of lading will be retained as part of the permanent documentation.

When samples are transferred, the individuals relinquishing and receiving the samples will sign the COC form and record the date and time of transfer. The sample collector will sign the form in the first signature space. The only exception to this is the shipment of samples via commercial carriers. Because sample containers are sealed with the COC record inside prior to delivery to the carrier, the custody signature will be that of the individual taking possession of the samples from the carrier at its final destination. Each person taking custody will observe whether the shipping container is correctly sealed and in the same condition as noted by the previous custodian; deviations will be noted on the appropriate section of the COC record.

A designated sample custodian at the laboratory will accept custody of the shipped samples, verify the integrity of the custody seals, and certify that the sample identification numbers match those on the COC record. The custodian will then enter sample identification number data into a bound logbook, which is arranged by a project code and station number. If containers arrive with broken custody seals, the laboratory will note this on the COC record and immediately notify the sampler who will, in turn, notify the QA manager and the LAI project manager/project coordinator.

6.4 Analytical Methods

Laboratory methods and target RLs for all potential analyses of soil, water, and soil gas are summarized in Tables 3 and 4. Samples collected and analyzed as part of the RI/FS will be reported to the MDL as necessary to meet project-specified SLs.

For all groundwater analyses except dissolved metals, any suspended material in the sample will be allowed to settle and the sample will not be agitated prior to analysis of the supernatant. For the dissolved metals analyses, the samples will be filtered in the field to remove any suspended material.

Sample containers, preservation, and holding times are provided in Table 5.

6.5 Quality Control

Field and analytical laboratory control samples will be collected to evaluate data precision, accuracy, representativeness, comparability, completeness, bias, and sensitivity of the analytical results for this investigation. The quality control samples and the frequency at which they will be collected and/or analyzed by matrix and analysis is summarized in Table 2. The evaluation of these quality control samples is further discussed in Section 8.

6.6 Instrument/Equipment/Consumables

To ensure that field measurement is accomplished accurately, field equipment undergoes routine maintenance and calibration as described below.

6.6.1 Testing, Inspection, and Maintenance

LAI performs routine inspections and preventive maintenance (parts replacement and cleaning) for all pieces of field equipment in our supply and equipment room. Maintenance activities are conducted by our field technicians, who are specifically trained in the use, operation, and maintenance of the equipment. All field equipment used during this project, which may include water level indicators, photoionization detectors (PIDs), and water field parameter meters (e.g., pH), will be cleaned and decontaminated prior to use. Each piece of equipment will be inspected and tested to ensure proper working function and facilitate replacement or repair of broken or non-operational components. Extra batteries will be included in the equipment cases or in field vehicles for replacing dead batteries during field work. Extra disposables will be packed for equipment requiring disposables for use, such as ferrous iron kits.

Field equipment is maintained by the field equipment manager. Field staff continually notify the field equipment manager when equipment maintenance is needed. This system ensures the equipment is maintained and working for the next field project. Equipment will be repaired or replaced, as needed.

Meters used to make field measurements will be further inspected and tested during calibration, as described in the next section.

6.6.2 Calibration and Frequency

All field equipment are calibrated according to the manufacturers' guidelines and recommendations. If a PID is used during this project, it will be calibrated on a daily basis according to the manufacturer's specifications. The PID preferred by LAI field personnel uses a 10.2-eV (electron volt) probe and is calibrated using a manufacturer-supplied standard gas (isobutylene, equivalent to 34 parts per million benzene). Similarly, water field parameter meters will be calibrated at the start of each sampling day with laboratory-prepared calibration standards within the range of the anticipated measurement. An instrument will also be recalibrated at any time an anomalous reading suggests instrument imprecision or inaccuracy.

6.6.3 Inspection/Acceptance of Supplies and Consumables

Supplies are ordered and maintained by the field equipment manager. Disposables and consumables include nitrile gloves, Ziploc® bags for sample ice, field test kits, and polyethylene tubing.

6.7 Data Management

All laboratory analytical results, including QC data, will be submitted electronically. Electronic formats will include a PDF file of the laboratory report, and electronic data deliverable (EDD) files that will be uploaded directly to an Environmental Quality Information Systems (EQuIS) database; the data management team will supply the required format for the EDDs. EQuIS EDDs will be provided by the laboratory in the EFWEDD format (also known as EQuIS 4-File), using LAI valid values. After validation of the data, any applicable qualifiers will be added to the database.

Field data (groundwater field parameter data and water levels measurements) will be entered into cumulative Excel® spreadsheets and/or the EQuIS database. Data will be verified to determine all entered data are correct and without omissions and errors.

Field notes, including field reports, sampling forms, survey forms, test pit logs, boring logs, and well construction diagrams, will be maintained in the project files. Survey notes will be reduced to provide coordinates and elevations that will be uploaded to the database.

Level II laboratory reports will include the following:

- Case narrative, including adherence to prescribed protocols, non-conformity events, corrective measures, and/or data deficiencies (including initial and continuing instrument calibrations, and explanations for any missed target RLs)
- Sample analytical results
- Surrogate recoveries
- Matrix spike/matrix spike duplicate results
- Blank spike/blank spike duplicate results
- Laboratory duplicates
- Blank results
- Sample custody (including signed COC records, and laboratory sample receipt forms)
- Analytical responsibility.

7.0 ASSESSMENT AND OVERSIGHT

This section describes assessment and oversight.

7.1 Assessment and Response Actions

Assessments during implementation of the project will include daily communication and updates during field work and data quality review by the LAI project manager/project coordinator and field staff. Response actions to assessed issues will be coordinated between the LAI project manager, field staff, the project manager for Snohomish County Public Works, and involved subcontractors, as appropriate. Data management assessment activities are discussed in greater detail in Section 8.2.4.

If any project non-conformance is considered significant or requires special expertise, corrective action(s) may include the following:

- Reanalyzing the samples, if holding times can be met
- Resampling and analyzing
- Evaluating and amending sampling and analytical procedures
- Accepting data and acknowledging the level of uncertainty or inaccuracy by flagging the data.

8.0 DATA VALIDATION AND USABILITY

This section describes data validation and usability.

8.1 Data Review, Verification, and Validation

All RI data will be verified and validated to determine that the results are acceptable and meet the quality objectives described in Section 3.

Validation of the data will be performed by a data validator with guidance from applicable portions of the National Functional Guidelines for Organic Data Review (EPA 2016b) and the National Functional Guidelines for Inorganic Data Review (EPA 2016a).

All data generated as part of the RI will undergo a Level IIA verification and validation.

EPA Level IIA-equivalent verification and validation elements are presented in Table 6 and will include the following:

- Verification that the laboratory data package contains all necessary documentation (including COC records; identification of samples received by the laboratory; date and time of receipt of the samples at the laboratory; sample conditions upon receipt at the laboratory; date and time of sample analysis; and, if applicable, date of extraction, definition of laboratory data qualifiers, all sample-related QC data, and QC acceptance criteria)
- Verification that all requested analyses, special cleanups, and special handling methods were conducted
- Verification that QC samples were analyzed per the method and frequency specified in the QAPP
- Evaluation of sample holding times
- Evaluation of QC data compared to acceptance criteria, including field QC samples (field duplicates, trip blanks, and/or equipment blanks) and laboratory QC samples (method blanks, surrogate recoveries, laboratory duplicate and/or replicate results, and LCS results)
- Verification that RLs for target analytes are at or below the target RLs specified in the QAPP.

In the event that a portion of the data is outside the DQO limits or the EPA guidance (EPA 2016a, b), or sample collection and/or documentation practices are deficient, corrective action(s) will be initiated. Corrective action, as described in Section 7.1, may include any of the following:

- Rejection of the data and resampling
- Qualification of the data
- Modified field and/or laboratory procedures.

8.2 Verification and Validation Methods

The processes that will be used to verify and validate data are described in the sections below.

8.2.1 Data Verification Methods

This section describes data verification methods.

8.2.1.1 Chain of Custody

COC forms will be reviewed by field personnel upon completion of sampling, who will verify information against the packed sample coolers they represent. A copy of the COC form will be retained in the electronic project files, and the original and remaining copies will be taped inside the cooler for delivery to the analytical laboratory.

8.2.1.2 Corrective Actions

The corrective action process may be initiated by any project team member. The process consists of identifying a problem, acting to eliminate the problem, documenting the corrective action, monitoring the effectiveness of the corrective action, and verifying that the problem has been sufficiently addressed. The LAI field lead will be responsible for correcting and resolving situations in the field that may result in non-compliance with the QAPP. Corrective measures identified by the field lead will be immediately documented in the field notes. Examples of corrective actions for field measurements may include: repetition of a measurement to check the error, check for proper adjustments for ambient conditions, check of batteries, recalibration, replacement of instruments, revisions to COCs forms, and (if necessary) stop work. Laboratory project managers are responsible for ensuring that corrective action processes as identified in their quality systems manuals, SOPs, and this QAPP are followed. The laboratory project manager is responsible for notifying the LAI quality manager of any non-conformance. If a corrective action is initiated at the laboratory, it shall be narrated in the laboratory data package. Technical staff will be responsible for reporting any QA non-conformance or suspected deficiencies they identify to the LAI project manager, who will in turn notify the LAI QA manager. The LAI QA manager is responsible for assessing the suspected deficiency or non-conformance and its potential to impact data quality.

If corrective actions are required, a copy of the documented corrective action taken will be maintained in the electronic project files. At the completion of the sampling event, the LAI QA officer and the LAI project manager will ensure all appropriate corrective actions have been taken and that the corrective action reports have been included in the electronic project files; if corrective actions have not been taken, the project manager will ensure action is taken.

8.2.1.3 Field Notes

Field notes will be reviewed internally and placed in the electronic project files.

8.2.1.4 Analytical Data Packages

All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.

All laboratory data packages, with the exception of waste characterization samples, will be verified by a data validator who is not associated with the collection or analysis of samples, interpretation of sample data, or with any decision-making process within the scope of the investigation.

The data validator will conduct an EPA Level IIA-equivalent validation and verification, which will be performed with guidance from applicable portions of the National Functional Guidelines for Organic Data Review (EPA 2016b) and the National Functional Guidelines for Inorganic Data Review (EPA 2016a). Additional information regarding the data validation process is provided in the following sections.

8.2.2 Data Validation Methods

Validation of the analytical data will include the criteria listed below. Validation procedures will be followed to ensure data are evaluated properly, completely, and consistently for use in meeting DQOs.

The data validator (unless noted otherwise) will complete the following:

- Data deliverables: Ensure all required verified information on sampling and analysis has been made available as part of data validation (see Section 8.2.1; this also includes associated planning documents [i.e., work plan, SAP, or QAPP]).
- Analytes: Ensure the required list of analytes was reported as specified in the planning documents.
- COC: Review the COC form for traceability of the data from sample collection through to data reporting.
- Holding times: Ensure samples were analyzed within specified holding times (i.e., method, procedure, or planning document). If holding times were not met, confirm the laboratory has documented any deviations and made appropriate notifications to the project team, and that approval to proceed was received prior to analysis.
- Sample handling: Ensure sample handling, receipt, and storage procedures were followed, with any deviations documented.
- Sampling methods and procedures: Establish that required sampling methods were used and any deviations documented. Ensure the sampling procedures and field measurements met performance criteria and any deviations were documented.
- Field transcription: Authenticate transcription accuracy of field data (i.e., from field forms to report tables).
- Analytical methods and procedures: Establish that required analytical methods were used, with any deviations documented. Ensure QC samples met performance criteria, with any deviations documented.
- Data qualifiers: Determine laboratory data qualifiers were defined and applied as specified (i.e., method, procedure, or planning document).

- Laboratory transcription: Authenticate accuracy of transcription of analytical data (i.e., instrument to the Laboratory Information Management System, or laboratory notebook to reporting form).
- Standards: Determine that standards are traceable and meet requirements (method, procedure, or planning document).
- Communication: Confirm required communication procedures were followed by field and/or laboratory personnel.
- Audits: Review laboratory audit reports, accreditation, and certification records for the laboratory's performance on specific methods; review field forms to verify compliance with work plan and QAPP procedures.

8.2.3 Data Validation Review and Data Qualification

For Level IIA data validation, data quality will be assessed by comparing QC parameters to the appropriate criteria (i.e., limits) as specified in the planning documents (i.e., work plan, SAP, QAPP).

Analytical data may be qualified based on the data validation review. Qualifiers will be consistent with applicable EPA national functional guidelines and will be used to provide data users with an estimate of the level of uncertainty associated with the qualified result.

Data validation results will be evaluated with respect to assigned qualifiers to determine any data usability issues. The following qualifiers may be assigned during the data validation process:

J	Indicates the analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analyte has been "tentatively identified" or "presumptively identified" as present and the associated numerical value is the estimated concentration in the sample.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The objectives, evaluations, and actions employed during the data validation process will be guided by EPA national functional guidelines. Laboratories will be permitted to provide CLP-like forms in lieu of original CLP forms. The data validation criteria will not strictly adhere to national functional guidelines, but will also take into consideration method criteria for preservation and holding times; laboratory-specified criteria for surrogate, laboratory control samples, laboratory duplicates, and matrix spikes; and the data validator's professional judgment.

9.0 PROJECTS USING EXISTING DATA

Since the TECT Aerospace RI/FS is an ongoing program, secondary data may be used to evaluate performance and concentration trends. Historical data will be considered usable for the decisions being made on this project, especially in light of the effort to identify and resolve data gaps.

10.0 USE OF THIS QUALITY ASSURANCE PROJECT PLAN

This Quality Assurance Project Plan has been prepared for the exclusive use of Snohomish County Public Works and applicable regulatory agencies for specific application to the former Paine Field TECT Aerospace Leasehold. No other party is entitled to rely on the information, conclusions, and recommendations included in this document without the express written consent of LAI. Further, the reuse of information, conclusions, and recommendations provided herein for extensions of the project or for any other project, without review and authorization by LAI, shall be at the user's sole risk. LAI warrants that within the limitations of scope, schedule, and budget, our services have been provided in a manner consistent with that level of care and skill ordinarily exercised by members of the profession currently practicing in the same locality under similar conditions as this project. We make no other warranty, either express or implied.

11.0 REFERENCES

ANSI/ASQC. 1994. American National Standard - Quality Systems - Model for Quality Assurance in Design, Development, Production, Installation, and Servicing. American Society for Quality. August 1. https://law.resource.org/pub/us/cfr/ibr/002/asqc.q9001.1994.pdf.

APHA, AWWA, and WEF. 1998. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, American Water Works Association, and Water Environment Federation. January 1.

Ecology. 2004. Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies. Publication No. 04-03-030. Washington State Department of Ecology. July. https://fortress.wa.gov/ecy/publications/summarypages/0403030.html.

EPA. 1974. Methods for Chemical Analysis of Water and Wastes. Publication No. EPA-625 / 6-74-003. Office of Technology Transfer, US Environmental Protection Agency.

EPA. 1999. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods; Third Edition; Final Update III-A. EPASW-846.3-3a. 3rd ed. Solid Waste and Emergency Response, US Environmental Protection Agency. March.

EPA. 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5. EPA/240/B-01/003. US Environmental Protection Agency. March.https://www.epa.gov/sites/production/files/2016-06/documents/r5-final_0.pdf.

EPA. 2002. Guidance for Quality Assurance Project Plans, EPA QA/G-5. EPA/240/R-02/009. US Environmental Protection Agency. December. https://www.epa.gov/sites/production/files/2015-06/documents/g5-final.pdf.

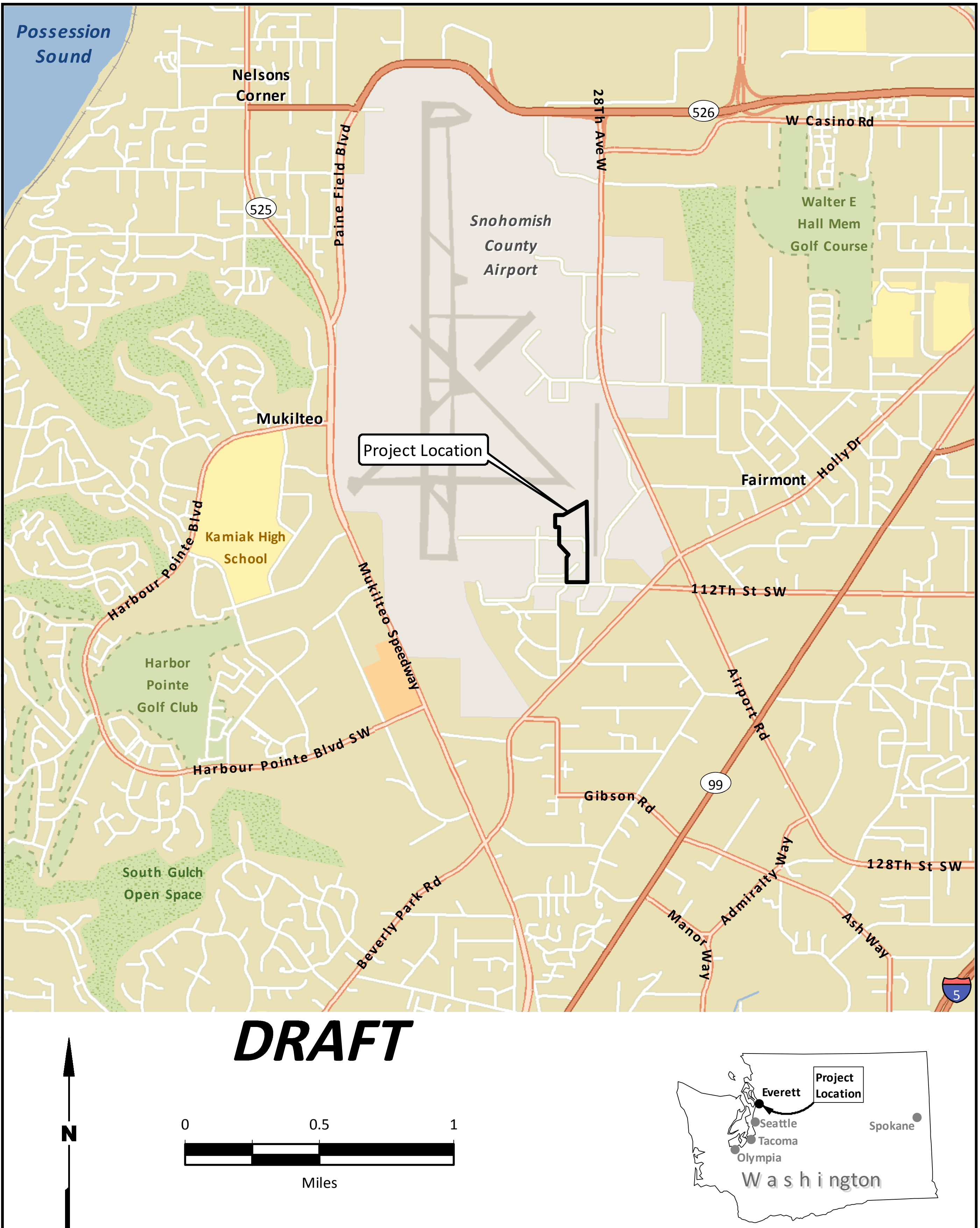
EPA. 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. OSWER No. 9200.1-85; EPA 540-R-08-005. US Environmental Protection Agency. January 13.

EPA. 2016a. National Functional Guidelines for Inorganic Superfund Methods Data Review. EPA-540-R-2016-001 OLEM 9355.0-133. US Environmental Protection Agency. September.

EPA. 2016b. National Functional Guidelines for Superfund Organic Methods Data Review. EPA-540-R-2016-002 OLEM 9355.0-134. US Environmental Protection Agency. September.

Johnson, Gary L. 1994. ANSI/ASQC E4: Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs. American Society of Quality Control. http://asq.org/ee/2003/06/energy-and-environmental-division-newsletter-v23-i02-feature-article-ansi-asqc-e4-1994-a-quali.html?shl=088571.

Snohomish County. "Permitting and Land Use Maps." https://snohomishcountywa.gov/2467/Permitting-and-Land-Use-Maps.



Data Source: Esri 2012

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LANDAU ASSOCIATES

TECT Aerospace Everett, Washington	**Vicinity Map**	Figure B-1

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Legend

▭ Approximate Site Boundary

Note

1. Black and white reproduction of this color original may reduce its effectiveness and lead to incorrect interpretation.

0 150 300

Scale in Feet

Data Sources: AGI 1999; Landau Associates 2006; Esri World Imagery.

Landau Associates

TECT Aerospace Everett, Washington	**Site Plan**	Figure B-2

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Table B-1
Key Personnel Project Responsibilities
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Name	Title/Role	Organization Affiliation	Responsibilities
Andrew Rardin	Project Manager	Snohomish County Public Works	Manages the project for Snohomish County.
Gerald Ninteman	Consultant Project Manager	Landau Associates	Supervises and coordinates all work for the project. These responsibilities include project planning and execution, scheduling, staffing, data evaluation, report preparation, subcontracts, and managing deliverables.
Danille Jorgensen	Quality Assurance Officer	Landau Associates	Oversees and directs quality assurance (QA) reviews for the project, including periodic reports, analytical program requirements, and schedules before submittal to EPA for review and comment. Responsible for inputting all field data and the maintenance of the database.
Kristi Schultz	Data Specialist	Landau Associates	Reviews laboratory analytical data and provides data validation. Has oversight responsibility for management and integrity of the data.
Chris Kimmel	Site Health & Safety Manager	Landau Associates	Responsible for review and implementation of the Health and Safety Plan (HASP).
Rick Bagan	Project Manager/ Project Coordinator	ALS Global Laboratories Everett	Manages laboratory analysis and reporting.
Sue Anderson	Project Manager/ Project Coordinator	ALS Global Laboratories Simi Valley	Manages laboratory analysis and reporting.

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Table B-2
Data Quality Objectives
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

DQI	QC Sample or Activity Used to Assess MQO	MQO	Frequency	Sampling or Analytical DQI
Soil Samples Analyzed for Gasoline-Range Petroleum Hydrocarbons by Method NWTPH-Gx				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <35%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank, Trip Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Soil Samples Analyzed for Diesel- and Motor Oil-Range Petroleum Hydrocarbons, and Jet Fuel by Method NWTPH-Dx				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <35%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Soil Samples Analyzed for Total Metals by EPA Methods 6020A and 7471A/7196				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Accuracy	LCS	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/Laboratory Duplicate	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <35%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Soil Samples Analyzed for Volatile Organic Compounds by EPA Method 8260C/8011				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Bias/Contamination	Method Blank, Trip Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S

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Table B-2
Data Quality Objectives
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

DQI	QC Sample or Activity Used to Assess MQO	MQO	Frequency	Sampling or Analytical DQI
Soil Samples Analyzed for Polychlorinated Biphenyls by EPA Method 8082A				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <35%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations > 1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Soil Samples Analyzed for Total Organic Carbon by ASTM Method D4129-05 Mod				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Accuracy	LCS	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/Laboratory Duplicate	Recoveries within laboratory-specified control limits	1 per 10 samples or two per analytical batch	S&A
Precision	Field Duplicates	RPD <35%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Gasoline-Range Petroleum Hydrocarbons by Method NWTPH-Gx				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank, Trip Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Diesel- and Motor Oil-Range Petroleum Hydrocarbons, and Jet Fuel by Method NWTPH-Dx				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S

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Table B-2
Data Quality Objectives
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

DQI	QC Sample or Activity Used to Assess MQO	MQO	Frequency	Sampling or Analytical DQI
Water Samples Analyzed for Total or Dissolved Metals by EPA Methods 6010C and 7471A/7196				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Accuracy	LCS	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/Laboratory Duplicate	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Volatile Organic Compounds by EPA Method 8260C/8011				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Bias/Contamination	Method Blank, Trip Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Semivolatile Organic Compounds by EPA Method 8270				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Nitrate or Sulfate by EPA Method 300.0				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Accuracy	LCS	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/Laboratory Duplicate	Recoveries within laboratory-specified control limits	1 per 10 samples or two per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S

Table B-2
Data Quality Objectives
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

DQI	QC Sample or Activity Used to Assess MQO	MQO	Frequency	Sampling or Analytical DQI
Water Samples Analyzed for Total Organic Carbon by Method SM 5310C				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Accuracy	LCS	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/Laboratory Duplicate	Recoveries within laboratory-specified control limits	1 per 10 samples or two per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Water Samples Analyzed for Natural Attenuation Parameters (Methane, Ethane, Ethene, Acetylene) by Method RSK-175				
Representativeness	Cooler Temperature	<6°C	All project samples	S
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD and MS/MSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Method performance for matrix, bias	MS/MSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	S&A
Precision	Field Duplicates	RPD <20%	1 per 20 samples or one per analytical group	S&A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S
Air Samples Analyzed for Volatile Organic Compounds by EPA Method TO-15				
Bias	Surrogates	Recoveries within laboratory-specified control limits	All project and QA samples	A
Accuracy	LCS/LCSD	Recoveries within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Precision	LCS/LCSD	RPDs within laboratory-specified control limits	1 per 20 samples or one per analytical batch	A
Bias/Contamination	Method Blank	Target analytes not detected at concentrations >1/2 the RL	1 method blank per 20 samples, 1 every 12 hours, or 1 per analytical batch	S&A
Analytical Completeness	Number of usable (not rejected) results out of total number of results	90%	NA	S&A
Field Completeness	Number of samples collected out of planned samples	95%	NA	S

Abbreviations/Acronyms:

A = analytical
% = percent
°C = degrees Celsius
DQI = data quality indicator
EPA = US Environmental Protection Agency
LCS = laboratory control spike
LCSD = laboratory control spike duplicate
LOQ = Limit of Quantitation
MQO = measurement quality objective
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
QA = quality assurance
QC = quality control
RL = reporting limit
RPD = relative percent difference
S = sampling
SIM = Selected Ion Monitoring

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Table B-3
Soil and Groundwater Targeted Reporting Limits
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Analyte	CAS No.	Soil (a)			Groundwater		
		Screening Level	RLs	Units	Screening Level	RLs	Units
Total Petroleum Hydrocarbons		Method ECY 97-602 NWTPH-Gx modified			Method ECY 97-602 NWTPH-Gx modified		
Gasoline Range	86290-81-5	30	3	mg/kg	800/1,000	50	µg/L
		Method ECY 97-602 NWTPH-Dx modified			Method ECY 97-602 NWTPH-Dx modified		
Diesel Range	TPHDIESELONLY	2,000	25	mg/kg	500	130	µg/L
Motor Oil Range	TPHOILONLY	2,000	50	mg/kg	500	250	µg/L
Total Metals		Method 6020A/7471A/7196					
Arsenic	7440-38-2	7	0.5	mg/kg	--	--	--
Cadmium	7440-43-9	1	0.5	mg/kg	--	--	--
Chromium, Total	7440-47-3	42	0.5	mg/kg	--	--	--
Chromium III (calculated)	16065-83-1	100	--	mg/kg	--	--	--
Chromium, Hexavalent	18540-29-9	0.926	5	mg/kg	--	--	--
Lead	7439-92-1	150	0.5	mg/kg	--	--	--
Mercury	7439-97-6	0.105	0.02	mg/kg	--	--	--
Dissolved Metals					Method 200.8/245.1/7196		
Arsenic	7440-38-2	--	--	--	5	1	µg/L
Cadmium	7440-43-9	--	--	--	5	1	µg/L
Chromium, Total	7440-47-3	--	--	--	100	2	µg/L
Chromium III (calculated)	16065-83-1	--	--	--	100	--	µg/L
Chromium, Hexavalent	18540-29-9	--	--	--	48	10	µg/L
Lead	7439-92-1	--	--	--	15	1	µg/L
Mercury	7439-97-6	--	--	--	2	0.2	µg/L
Volatile Organic Compounds		Method EPA 8260C			Method EPA 8260C		
1,1,1,2-Tetrachloroethane	630-20-6	38,000	10	µg/kg	1.68	0.5	µg/L
1,1,1-Trichloroethane	71-55-6	84.3	10	µg/kg	200	2	µg/L
1,1,2,2-Tetrachloroethane	79-34-5	0.080	1.5	µg/kg	0.5	0.5	µg/L
1,1,2-Trichloroethane	79-00-5	0.278	1.5	µg/kg	0.768	0.5	µg/L
1,1-Dichloroethane	75-34-3	2.61	1.5	µg/kg	7.68	2	µg/L
1,1-Dichloroethene	75-35-4	2.46	1.5	µg/kg	7	2	µg/L
1,2,4-Trimethylbenzene	95-63-6	--	10	µg/kg	--	2	µg/L
1,2-Dichloroethane	107-06-2	1.5	1.5	µg/kg	0.481	0.02	µg/L

Table B-3
Soil and Groundwater Targeted Reporting Limits
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Analyte	CAS No.	Soil (a)			Groundwater		
		Screening Level	RLs	Units	Screening Level	RLs	Units
1,2-Dichloropropane	78-87-5	1.67	1.5	µg/kg	1.22	0.5	µg/L
1,3,5-Trimethylbenzene	108-67-8	800,000	10	µg/kg	80	2	µg/L
2-Butanone	78-93-3	48,000,000	50	µg/kg	4,800	10	µg/L
2-Hexanone	591-78-6	--	50	µg/kg	--	10	µg/L
4-Isopropyltoluene	99-87-6	--	10	µg/kg	--	2	µg/L
4-Methyl-2-Pentanone (MIBK)	108-10-1	6,400,000	50	µg/kg	640	10	µg/L
Acetone	67-64-1	2,070	50	µg/kg	7,200	25	µg/L
Benzene	71-43-2	0.277	1.5	µg/kg	0.795	0.5	µg/L
Carbon Disulfide	75-15-0	266	10	µg/kg	800	2	µg/L
Carbon Tetrachloride	56-23-5	0.274	1.5	µg/kg	0.625	0.5	µg/L
Chloroethane	75-00-3	--	10	µg/kg	--	2	µg/L
Chloroform	67-66-3	0.479	1.5	µg/kg	1.41	0.5	µg/L
cis-1,2-Dichloroethene	156-59-2	5.15	1.5	µg/kg	16	2	µg/L
Ethylbenzene	100-41-4	343	10	µg/kg	700	2	µg/L
Ethylene Dibromide (1,2-Dibromoethane)	106-93-4	500	5	µg/kg	0.02	0.01	µg/L
Isopropylbenzene	98-82-8	8,000,000	10	µg/kg	800	2	µg/L
Methyl T-Butyl Ether	1634-04-4	7.23	1.5	µg/kg	24.3	2	µg/L
Methylene Chloride	75-09-2	1.48	1.5	µg/kg	5	5	µg/L
Naphthalene	91-20-3	236	10	µg/kg	160	2	µg/L
n-Propylbenzene	103-65-1	8,000,000	10	µg/kg	800	2	µg/L
sec-Butylbenzene	135-98-8	8,000,000	10	µg/kg	800	2	µg/L
Tetrachloroethene	127-18-4	2.76	1.5	µg/kg	5	2	µg/L
Toluene	108-88-3	273	10	µg/kg	640	2	µg/L
trans-1,2-Dichloroethene	156-60-5	32.5	10	µg/kg	100	2	µg/L
Trichloroethene	79-01-6	0.206	1.5	µg/kg	0.54	0.5	µg/L
Vinyl Chloride	75-01-4	0.009	0.05	µg/kg	0.029	0.2	µg/L
Total Xylenes	1330-20-7	831	20	µg/kg	1,600	4	µg/L
Conventional Parameters					Method EPA 300.0		
N-Nitrate	14797-55-8	--	--	--	10,000	153	µg/L
Sulfate	14808-79-8	--	--	--	--	260	µg/L
		ASTM Method D4129-05 Mod			Method SM 5310C		
Total Organic Carbon	877-24-7	--	0.10	%	--	500	µg/L

Table B-3
Soil and Groundwater Targeted Reporting Limits
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Analyte	CAS No.	Soil (a)			Groundwater		
		Screening Level	RLs	Units	Screening Level	RLs	Units
Natural Attenuation Parameters					Method RSK-175		
Methane	74-82-8	--	--	--	--	10	µg/L
Ethane	74-84-0	--	--	--	--	10	µg/L
Ethene	74-85-1	--	--	--	--	10	µg/L
Semivolatile Organic Compounds					Method EPA 8270		
1,4-Dioxane	123-91-1	--	--	--	0.44	0.04	µg/L
Volatile Organic Compounds		EPA Method 8011			EPA Method 8011		
Ethylene Dibromide	106-93-4	0.5	0.005	mg/kg	0.02	0.01	µg/L
PCBs		EPA Method 8082A					
Aroclor 1016	12674-11-2	5.6	0.10	mg/kg	--	--	--
Aroclor 1242	53469-21-9	--	0.10	mg/kg	--	--	--
Aroclor 1248	12672-29-6	--	0.10	mg/kg	--	--	--
Aroclor 1254	11097-69-1	0.5	0.10	mg/kg	--	--	--
Aroclor 1260	11096-82-5	0.5	0.10	mg/kg	--	--	--
Aroclor 1221	11104-28-2	--	0.10	mg/kg	--	--	--
Aroclor 1232	11141-16-5	--	0.10	mg/kg	--	--	--
Aroclor 1268	11100-14-4	--	0.10	mg/kg	--	--	--
Total PCBs	--	0.5	--	mg/kg	--	--	--

Abbreviations and Acronyms:

CAS = Chemical Abstracts Services
EPA = US Environmental Protection Agency
µg/kg = micrograms per kilogram
µg/L = micrograms per liter
MDL = method detection limit
mg/kg =milligrams per kilogram
mg/L = milligrams per liter

-- = not applicable
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyls
RL = reporting limit
SIM = selected ion monitoring
SL = screening level

Notes:

(a) Soil results and associated laboratory reporting limits will be reported on a dry weight basis

Table B-4
Soil Gas Targeted Reporting Limits
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Analyte	CAS No.	Shallow Soil Gas Screening Level	RLs	Units
Volatile Organic Compounds			EPA TO-15	
1,1,1-Trichloroethane	71-55-6	76,200	1.4	µg/m^3
1,1-Dichloroethane	75-34-3	52.1	1.3	µg/m^3
Benzene	71-43-2	10.7	1.3	µg/m^3
Tetrachloroethene	127-18-4	321	1.3	µg/m^3
Trichloroethene	79-01-6	12.3	1.3	µg/m^3
Vinyl chloride	75-01-4	9.33	1.3	µg/m^3

Abbreviations and Acronyms:

CAS = Chemical Abstracts Services
EPA = US Environmental Protection Agency
µg/m^3 = micrograms per cubic meter

Table B-5
Sample Containers, Preservatives, and Holding Times
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Matrix	Method	Container	Preservative	Holding Time (a)	Laboratory Performing Analyses
Soil	Gasoline-range Petroleum Hydrocarbons by NWTPH-Gx	4 oz + 5035 methanol vial	<6^{o}C	14	ALS Global Everett
Groundwater	Gasoline-range Petroleum Hydrocarbons by NWTPH-Gx	2 x 40-mL glass	Add HCl to pH<2; <6^{o}C	14	ALS Global Everett
Soil	Diesel- and Oil-range Petroleum Hydrocarbons by NWTPH-Dx	4 oz	<6^{o}C	14 days/40 days	ALS Global Everett
Groundwater	Diesel- and Oil-range Petroleum Hydrocarbons by NWTPH-Dx	500-mL amber glass	<6^{o}C	7 days/40 days	ALS Global Everett
Soil	Total Metals by EPA 6020A (7471A for mercury)	4 oz amber glass	<6^{o}C (mercury only)	180 (mercury 28 days)	ALS Global Everett
Soil	Hexavalent Chromium by EPA 7196	4 oz polyethylene or glass	<6^{o}C	30 days to extraction then 24 hours for analysis	ALS Global Everett
Groundwater	Dissolved Metals by EPA Method 200.8 (245.1 for mercury)	500 mL plastic	If field filtered, HNO_3 to pH <2; <6^{o}C	180 (mercury 28 days)	ALS Global Everett
Groundwater	Hexavalent Chromium by EPA 7196	500 mL polyethylene or glass	<6^{o}C	24 hours	ALS Global Everett
Soil	VOCs by EPA Method 8260/8011	4 oz, + 5035 methanol vila and two stirbar vials	HCl to pH<2; <6^{o}C	14 days freeze stirbar vials within 48 hours	ALS Global Everett
Groundwater	VOCs by EPA Method 8260/8011	3 x 40-mL glass	HCl to pH<2; <6^{o}C	14 days (7 days pH >2)	ALS Global Everett
Groundwater	Nitrate by EPA Method 300.0	500 mL polyethylene	<6^{o}C	48 hours	ALS Global Everett

Table B-5
Sample Containers, Preservatives, and Holding Times
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

Matrix	Method	Container	Preservative	Holding Time (a)	Laboratory Performing Analyses
Groundwater	Sulfate by EPA Method 300.0	500 mL polyethylene	<6°C	28	ALS Global Everett
Soil	Total Organic Carbon by ASTM D4129-05 Modified	4 oz amber glass	<6°C	28	ALS Global Kelso
Groundwater	Total Organic Carbon by Method SM 5310C	500 mL amber glass	H_2SO_4 to pH <2; <6°C	28	ALS Global Kelso
Groundwater	Natural Attenuation Parameters (methane, ethane, ethene, acetylene) by Method RSK-175	3 x 40-mL amber glass	HCL to pH <2; <6°C	14	ALS Global Everett
Groundwater	1,4-Dioxane by EPA Method 8270M	2 x 1-L amber glass	<6°C	7 days/40 days	ALS Global Kelso
Soil	PCBs by EPA Method 8082A	4 oz polyethylene or glass	<6°C	365 days/40 days	ALS Global Everett

Acronyms/Abbreviations:

°C = degrees Celsius
EPA = US Environmental Protection Agency
g = gram
H_3PO_4 = Phosphoric acid
HCL = Hydrochloric acid
HNO_3 = nitric acid
L = liter
mL = milliliter
oz = ounces
cPAH = carcinogenic polycyclic aromatic hydrocarbor
PCB = polychlorinated biphenyl
SIM = selected ion monitoring
SVOC = semivolatile organic compound
VOC = volatile organic compound

Notes:

(a) Time from sample collection to extraction/time from sample extraction to analysis

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Table B-6
Data Validation Elements
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

QC Element	Evaluation Criteria	Qualification	Comments
Case Narrative	A case narrative shall be included with all laboratory packages.	Depending on issues presented in case narrative, additional qualification to the data may be warranted.	
Chain of Custody	A COC shall be included with all laboratory packages.	If discrepancies are noted on the COC, then the laboratory report may be revised to correct any issues.	
Preservation	Preservation conditions as noted in laboratory report are compared to method-specified requirements.	Depending on the preservation issue, data may be qualified as estimated (J/UJ) or rejected.	
Headspace	VOA vials should be free of headspace and air bubbles.	If sample was analyzed from a vial that contained headspace or bubbles, data will be qualified as estimated (J/UJ).	Applicable only to VOAs.
Sample Filtration	Samples that are field filtered shall be identified as such on the COC. Filtered metals will be reported as dissolved fraction.	If discrepancies are identified or problems with filtration are noted, then a revised lab report may be issued.	Applicable only to dissolved metals.
Holding Times	Holding times are compared to method-specified hold times.	If hold times are exceeded, then all results for the method are qualified as estimated (J/UJ). If hold times are grossly exceeded, then detected results are qualified as estimated (J) and no detected results are rejected (R).	

Table B-6
Data Validation Elements
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

QC Element	Evaluation Criteria	Qualification	Comments
Method Blanks	Detections of target analytes should be < RL for the analyte or < level of acceptable blank contamination specified in the QAPP	If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and between MDL and RL, raise result to RL and flag "U." If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and RL, flag "U." Apply method blank results to all samples in the same analytical batch.	
Field/Equipment Blanks	Detections of target analytes should be < RL for the analyte or < level of acceptable blank contamination specified in the QAPP	If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and between MDL and RL, raise result to RL and flag "U." If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and RL, flag "U." Apply field blank results to samples with same collection date; apply equipment blank results to samples associated with equipment.	

Table B-6
Data Validation Elements
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

QC Element	Evaluation Criteria	Qualification	Comments
Trip Blanks	Detections of target analytes should be < RL for the analyte or < level of acceptable blank contamination specified in the QAPP	If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and between MDL and RL, raise result to RL and flag "U." If sample result is <5x contaminant concentration (10x for common laboratory contaminants) and RL, flag "U." Apply trip blank results to samples shipped in the same cooler.	
LCS	Recoveries are compared to laboratory-specified QC limits.	If % is <10%, qualify detected results as estimated (J) and reject nondetected results. If %R is < laboratory-specified QC limits, qualify results as estimated (J/UJ). If %R is > laboratory-specified QC limits, qualify detected results as estimated (J).	
Surrogates	Recoveries are compared to laboratory-specified QC limits.	If % is <10%, qualify detected results as estimated (J) and reject nondetected results. If %R is < laboratory-specified QC limits, qualify results as estimated (J/UJ). If %R is > laboratory-specified QC limits, qualify detected results as estimated (J).	Not applicable for inorganics
MS	Recoveries are compared to laboratory-specified QC limits.	If % is <10%, qualify detected results as estimated (J) and reject nondetected results. If %R is < laboratory-specified QC limits, qualify results as estimated (J/UJ). If %R is > laboratory-specified QC limits, qualify detected results as estimated (J).	

DRAFT

Table B-6
Data Validation Elements
Quality Assurance Project Plan
Paine Field TECT Aerospace Leasehold
Everett, Washington

QC Element	Evaluation Criteria	Qualification	Comments
Laboratory Duplicate or MSD or LCSD	RPDs are compared to laboratory-specified QC limits.	If RPDs exceed laboratory-specified QC limits, then results for the sample that was analyzed in duplicate will be qualified as estimated (J/UJ).	
Dilutions	Results shall be reported within the calibration range of the instrument.	Results reported by the laboratory that are outside the calibration range of the instrument (E-qualified) will be marked as not reportable during data validation. The detected result that is within the calibration range is the reportable result. Nondetected results will be reported from the lowest dilution run.	
Field duplicates	RPDs should be <20% for aqueous samples and <50% for soil samples. For detected results <5 times their RLs, results should be within +- the RL.	RPD >20% waters (>35% soils), flag detected results “J.” Differences in concentrations > the RL, flag detected results “J.”	

Abbreviations and Acronyms:

% = percent
COC = chain of custody
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MDL = maximum detection limit
MS = matrix spike
MSD = matrix spike duplicate
QAPP = Quality Assurance Project Plan
QC = quality control
RL = reporting limit
RPD = relative percent difference
VOA = volatile organic analylsis

Notes:

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample

U = The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

ATTACHMENT B-1

Laboratory Standard Operating Procedures

ALS Environmental

930.0 Volatile Organics by GC/MS

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930.0 VOLATILE ORGANICS BY GC/MS

SOPID:	930.0 Volatile Organics by GC/MS	Rev. Number:	03.0	Effective Date:	3/1/2018

Approved By: _________________Glen Perry__________ Date: ______8/13/2018_

QA Manager – Glen Perry

Approved By: ____________________Rick Bagan______ Date: _______8/13/2018_

Laboratory Director – Rick Bagan

Archival Date:	____________	Doc Control ID#:	____________	Editor:	____________

Volatile Organics by GC/MS

1.0 Purpose

1.1 To define the procedure used to analyze for content of volatile organic compounds in environmental solid, liquid, or gas samples.

2.0 References

2.1 ALS Quality Assurance Manual (QAM).

2.2 Test Methods for Evaluating Solid Waste, USEPA-EMSL, SW-846, Method 8260C.

2.3 Hewlett Packard ChemStation and EnviroQuant users manuals.

3.0 Definitions

3.1 Method Blank – 5ml of analyte free water spiked with internal standards and surrogates at 20ng/mL. It is used to assess whether reagents used in the analysis are contaminated with any of the analytes of interest.

3.2 Blank Spike – A Method Blank spiked with internal standards, surrogates, and spike compounds at 10ng/ml (20ng/g for solids). Used to assess the effectiveness of the analytical technique in recovering the compounds of interest from a clean matrix. These are done in duplicate to provide a measure of laboratory precision.

3.3 Matrix Spike – An analytical sample spiked with internal standards, surrogates, and spike compounds at 10ng/ml (20ng/g for solids). Used to assess the effectiveness of the analytical technique in recovering the compounds of interest from an actual sample matrix. These are done in duplicate to provide a measure of laboratory precision and to assess the homogeneity of the matrix.

3.4 Trip Blank – A 40ml VOA vial filled with analyte free water at the laboratory and sent out with a set of containers to be used for sample collection. It is not opened in the field, but is returned to the laboratory with the samples. It is analyzed to assess the possibility of contamination of the samples by infiltration through the seals on the containers.

3.5 Standard Addition – The Archon autosampler is capable of adding a mixture of internal standards/surrogates to each sample before it is purged.

3.6 Extracted Ion Current Profile (EICP) – A plot of the intensity of a single mass versus time. The most common usage is to integrate a chromatographic peak by the characteristic mass of the compound involved.

3.7 Response Factor (RF) – The ratio of the area generated by integrating the EICP of the characteristic mass of a target analyte to the area generated by integrating the EICP of the characteristic mass of the relevant internal standard.

3.8 High Level 5035A – A soil sample prepared using the 5035A guidelines for analysis of volatile organic compounds in a solid matrix. High level samples are preserved by methanol in the field.

3.9 Low Level 5035A – A soil sample prepared using the 5035A guidelines for analysis of volatile organic compounds in a solid matrix. Low level samples are stored at -7C and must be frozen within 48 hours of sampling.

4.0 Apparatus and Materials

4.1 Purge and trap GC/MS analytical system consisting of the following:

4.1.1 Vial-based autosampler (Archon-type, Teledine Tekmar Atomix)

4.1.2 Purge and trap concentrator with Purge Trap K (Tekmar 2000/3000 series).

4.1.3 HP Gas Chromatograph (7890A or 7890B) with EPC and J&W 0.25mm DB-624 column (or equivalent).

4.1.4 HP Mass Selective detector (5975 or 5977).

4.1.5 IBM compatible PC running HP MS chemstation and Enviroquant software.

4.2 40ml VOA vials with caps and septa.

4.3 100mL volumetric flask.

4.4 10uL, 25uL, 50uL, 100uL, 1.0mL, 5.0mL and 10 mL gas tight syringes.

5.0 Reagents

5.1 Internal standard/surrogate solution – solutions containing the internal standards (pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5 and 1,4-dichlorobenzene-d4) and the surrogates (1,2-dichloroethane-d4, toluene-d8, p-bromofluoro-benzene) are prepared from commercially available stocks at 100ug/ml for each compound. Prepared in purge and trap grade methanol.

5.2 Calibration standard solution – solutions containing all compounds of interest are prepared from commercially available stocks at 100ug/ml for each compound. A second identical solution prepared at 100ug/ml from stocks obtained from a different source is used to verify calibration. Prepared in purge and trap grade methanol. Standard should be made at least every six months.

5.3 Spike solution - solutions containing the spike compounds (1,1-dichloroethene, benzene, trichloroethene, toluene, chlorobenzene) are prepared from commercially available stocks at 100ug/ml for each compound. Prepared in purge and trap grade methanol.

5.4 Volatile organic free deionized (DI) water: Drawn from Barnstead Nonopure water system.

5.5 Purge and trap grade methanol – high purity methanol, certified to be free of volatile analytes. Obtained commercially.

6.0 Sample Handling and Preservation

6.1 Water samples are collected in 40ml VOA vials with no headspace and preserved with HCl at pH<2.

6.2 Soil/solid samples are collected in 4oz. jars, packed tight to minimize headspace or collected by 5035A.

6.3 Air samples are collected in Tedlar bags.

6.4 Water samples and soil samples collected in jars are stored at 2 to 6C and must be analyzed within 14 days of collection; low level 5035A (direct sparge) samples are stored at -7 to -20C and must be analyzed within 14 days of collection; high level 5035A (methanol preserved) samples are stored at 2 to 6C and must be analyzed within 14 days of collection. Air samples are stored at room temperature and must be analyzed within 72 hours of collection.

7.0 Procedure – Normal daily operations consist of verifying MS tune, verifying continuing calibration, verifying instrument cleanliness, and analysis of samples. An initial calibration is performed only when necessary (see 8.2). Due to the complexity of the data system it is assumed that the operator has read and is familiar with the MS ChemStation and EnviroQuant manuals.

7.1 Instrument Operation - Systems will vary depending on manufacturer and model. The operator is assumed to be familiar with the automated sampling system and data acquisition software. The operator will set up the purge and trap system to automatically sample the appropriate amount of the matrix, add the appropriate amount and type of surrogate/internal standard, and follow the method-specified purge and trap procedure.

7.1.1 The automated sampling system will add 1uL of 100ug/mL Internal Standard/Surrogate Standard mixture to the water or soil sample before initiating purge.

7.1.2 The purge and trap system will Purge the sample for 11 minutes with the trap set at 40C. This may be followed by a Dry Purge step. The system will then preheat to 250C and Desorb for 1.5 minutes. The system will then Bake at 260C for 8 minutes.

7.1.3 When the purge and trap Desorbs the sample onto the GC/MS the instrument will automatically start. A delay before data acquisition (determined by the analyst) will avoid scanning the solvent peak and possibly damaging the filaments. The GC will ramp to a column-appropriate maximum temperate at a rate that will allow sufficient separation of target analytes to allow accurate quantization. The analyst will determine the appropriate split ratio and flow rate to optimize GC/MS response. The analyst will determine the appropriate threshold, sampling rate, and scan parameters to optimize GC/MS response.

7.2 Mass Spectrometer Tune.

7.2.1 The tune of the mass spectrometer must be verified before any standards or samples can be analyzed. The acquisition of a successful tune verification starts a 12 hour period during which all standards and samples must be analyzed. If an analysis cannot be done within this 12 hour window, then the tune must again be verified (thus starting a new 12 hour window).

7.2.2 50ng of p-bromofluorobenzene is introduced into the analytical system by running 5ml of a 10ng/ml solution of internal standard/surrogate. Once the data for p-BFB is acquired (retention time is ~16 minutes) the analyst evaluates the BFB using the Chemstation software and prints a hard copy for the instrument records.

7.3 Initial Calibration

7.3.1 Water analysis.

7.3.1.1 A 100ml volumetric flask is filled with analyte free water.

7.3.1.2 An appropriate amount of the 100ug/mL calibration standard is added to the 100mL volumetric flask to bring the concentration of the target compounds to the required level (1uL of standard would be added to create a 1ng/mL calibration standard, for example).

7.3.1.3 The analyst transfers the standard to a 40mL VOA vial (being careful to not leave any headspace) and discards the excess standard.

7.3.1.4 A minimum of 5 standards must be created for calibration by 8260 and it is strongly recommended that at least 7 standard levels be made. A typical calibration curve would have standards at the following levels: 1ng/mL, 2ng/mL, 5ng/mL, 10ng/mL, 15ng/mL, 20ng/mL and 30ng/mL.

7.3.1.5 The standards (VOA vials) are loaded into the autosampler and the autosampler is programmed to run the standards as water samples.

7.3.2 Soil analysis.

7.3.2.1 A 100ml volumetric flask is filled with analyte free water.

7.3.2.2 10uL of the 100ug/mL calibration standard is added to the 100mL volumetric flask to bring the concentration of the target compounds in the 100mL volumetric flask to 10ng/mL.

7.3.2.3 10mL of analyte free water is added to a 40mL VOA vial containing 5g of clean sand matrix (analyst may omit the 5g of clean sand if it can be demonstrated that the use of the blank matrix does not impact the quality of the calibration).

7.3.2.4 An appropriate amount of water is removed from the VOA vial and an appropriate amount of the 10ng/mL standard is added (1mL of water would be removed and 1mL of 10ng/mL standard would be added to create a 10ng/5g (2ng/g) calibration standard, for example).

7.3.2.5 For calibration standard levels above 20ng/g, 50uL of the 100ug/mL calibration standard is added to the 100mL volumetric flask to bring the concentration of the target compounds in the 100mL volumetric flask to 50ng/mL. All other steps remain the same.

7.3.2.6 A minimum of 5 standards must be created for calibration by 8260 and it is strongly recommended that at least 7 standard levels be made. A typical calibration curve would have standards at the following levels: 2ng/g, 4ng/g, 10ng/g, 20ng/g, 30ng/g, 40ng/g and 60ng/g.

7.3.3 Calibration table.

7.3.3.1 Once all of the standards have been analyzed, the quantitation report for each standard must be checked for errors. Some manual integration will be necessary since the automatic quantitation routines will miss some of the compounds in the low concentration standards and incorrectly integrate some compounds in the high concentration standards.

7.3.3.2 The analyst then loads the calibration curve into the Chemstation software and verifies that it meets the calibration requirements as specified in 8260C:

i. At least 5 calibration levels exist for each target analyte. Calibration levels may be removed at the high end or low end of the calibration for a target analyte provided at least 5 levels remain and no levels are removed between the high and low end of the calibration range (no 'holes' in the calibration, if you will). If the analyte is to be evaluated using a quadratic curve at least 6 calibration levels must exist.

ii. 90% of target analytes must have a %RSD equal to or less than 20 if the average of response factors is to be used as the curve fit.

iii. All target analytes must have a coefficient of determination (r^2) equal to or greater than 0.99 if a linear regression is to be used as the curve fit.

iv. All target analytes must have a coefficient of determination (r^2) equal to or greater than 0.99 if a quadratic regression is to be used as the curve fit. In addition the target analyte must have at least 6 calibration levels instead of a minimum of 5.

v. The 8260C Table 4 compounds must have the minimum response factors required by the method (see attached Table).

7.3.4 Once the calibration table is complete a midpoint (10ng/ml or 20ng/g) standard is prepared from stocks obtained from a different source. This standard is run and quantitated against the new initial calibration to verify the accuracy of the calibration. The calculated concentration of target analytes in the second source must match that of the initial calibration +/- 30%.

7.4 Continuing Calibration.

7.4.1 Once an acceptable tune has been obtained (see 7.2) a midpoint standard (10ng/ml or 20ng/g) is prepared (see 7.3.1 and 7.3.2), run and quantitated against the initial calibration.

7.4.2 80% of the target compounds must be within 20% difference of the initial calibration for the continuing calibration verification.

7.4.3 The 8260C Table 4 compounds must have the minimum response factors required by the method (see attached Table).

7.4.4 The response of the internal standards in the calibration verification must be within 50-200% of those of the midpoint of the calibration curve.

7.4.5 Target analyte retention times must be within 0.5 minutes of the retention times found in the midpoint of the calibration curve.

7.5 Method Blank.

7.5.1 Once an acceptable continuing calibration is obtained a method blank is run to verify cleanliness of the analytical system and reagents.

7.6 Quality Control Samples.

7.6.1 A Blank Spike and Blank Spike Duplicate must be run every day for Water/Air or Soil samples. A Matrix Spike and Matrix Spike Duplicate must be run for every batch of 20 Water/Air or Soil samples. The Laboratory Control Samples must contain all target compounds.

7.7 Analytical Samples.

7.7.1 Water samples are loaded directly into the autosampler.

7.7.2 Soil samples are prepared by weighing roughly 10 grams of sample into a VOA vial with a stir bar in it, adding 10mL of analyte free water, and capping the vial. These vials are then loaded into the autosampler.

7.7.3 Low level 5035A soil vials are weighed, the sample weight is calculated, and the vials are loaded directly into the autosampler.

7.7.4 For air samples a 50cc aliquot is removed from the Tedlar bag with a gas-tight syringe. The instrument is set up to run blank water samples and as the blank purges the air sample is injected into the concentrator at a rate of 100cc/minute.

7.7.4.1 Air samples may also be run in soil mode on the Archon by injecting a maximum of 25cc of sample into a VOA in which 10mL of analyte free water has already been added.

7.7.5 For high level 5035A samples and for samples which are inappropriate for direct purge and trap analysis, e.g. samples with very high concentrations of target analytes, oils and oily soils, etc., a methanol extraction step is included before analysis.

7.7.5.1 High level 5035A soil vials are weighed and the sample weight is calculated. Other samples are prepared as follows:

10g of the sample is weighed into a 40ml VOA vial and 10ml of purge and trap grade methanol is added. The sample/extract is agitated by placing it in a sonic bath for 5 minutes and the extract is then centrifuged to separate the methanol from the soil.

7.7.5.2 800ul of the methanol extract is transferred to a VOA vial filled with 40mL analyte free water.

7.7.5.3 The vial thus prepared with the extract is then run just like a water sample. QC acceptance criteria for soils are used to evaluate the results since the original matrix was a soil/solid.

7.7.5.4 After analysis the solvent volume must be adjusted based on the dry weight of the soil sample. For example a 5g soil sample extracted into 5mL of methanol found to be 80% solids would calculate final results based on a 4g sample size and 6mL of methanol/water extract.

8.0 Quality Control

8.1 Performance Criteria.

8.1.1 The acceptable p-bromofluorobenzene ion abundances are listed below:

mass 50 = 10-40% of mass 95
mass 75 = 30-60% of mass 95
mass 95 = 100% relative abundance (base peak)
mass 96 = 5-9% of mass 95
mass 173 = <2% of mass174
mass 174 = 50-100% of mass 95
mass 175 = 5-9% of mass 174
mass 176 = 95-101% of mass 174
mass 177 = 5-9% of mass 176

8.1.2 The limits for recovery of surrogates and matrix spikes as well as the limits for RPD in MS/MSD are published in “ALS Environmental Laboratories - Everett QC Sample Control Limits”.

8.2 Corrective Action.

8.2.1 If in any initial or continuing calibration one or more compounds listed in 8260C Table 4 fail to meet minimum response criteria the analytical system is in need of maintenance. Most problems with poor response are the fault of the purge and trap system. Only very rarely will the chromatographic system be at fault.

8.2.1.1 The Purge Trap K can be damaged by high concentrations of late gas range and early diesel range hydrocarbons. A trap which has been damaged in this fashion will lose efficiency trapping bromomethane, bromoform, and 1,2-dibromo 3-chloropropane. The only solution is to replace the trap.

8.2.1.2 Activity can occur in the gas lines of the concentrator which will cause dehydrohalogenation. If 1,1,2,2-tetrachloroethane response is low (especially if trichloroethene response is high at the same time) then this activity is present. Flush the entire sample transport path with a VERY weak (pH4-5) solution of HCl followed by organic free water.

8.2.1.3 A very common cause of poor response for the permanent gases is using standards which are too old. The first compound to be lost is dichlorodifluoromethane followed by chloromethane. Replace the standards with freshly made solutions.

8.2.2 If in a continuing calibration the response of more than 20% of the target analytes are different from that of the initial calibration by more than 20%, the calibration of the chromatographic system is suspect and the cause must be determined. Note that the 20% allowance of 8260C is a very generous and typically an ALS analyst should be expected to take corrective action if any analytes of concern recover outside of 20%.

8.2.2.1 If the standard solution used to make the continuing calibration is old, make up a fresh solution and try another continuing calibration.

8.2.2.2 If the tune of the mass spectrometer has changed significantly since the initial calibration was done the relative response of many compounds can be affected. The analyst should try to retune the mass spectrometer so that the ion abundances are the same (or nearly the same) as when the initial calibration was done and run another continuing calibration.

8.2.2.3 If the cause of the difference is not immediately apparent and there is no other reason to believe that the instrument is in need of maintenance, simply run a new initial calibration.

8.2.3 If the mass spectrometer becomes difficult or impossible to tune the ion source may be dirty. The instrument must be taken offline, the mass spectrometer must be vented, disassembled, and the ion source cleaned. If the instrument cannot be tuned with a clean source the ion optics are misaligned and professional maintenance is required.

8.2.4 If two or more surrogate recoveries in a sample analysis are outside recovery limits the sample must be reanalyzed. If the same surrogate(s) are still outside recovery limits it is considered to be due to the impact of the sample matrix and no further reanalysis is required. Note that an MS/MSD can fulfill this requirement.

8.2.5 If a sample has a result for one or more target compounds in excess of linear range the sample is diluted sufficiently to bring the result(s) into linear range and the diluted sample is reanalyzed.

9.0 Records Management

9.1 All tune, initial calibration and continuing calibration results are filed in storage boxes on site in the company store room for archival purposes.

9.2 All blank and sample results are submitted in the appropriate project folders along with a summary of the relevant quality control data.

9.3 A run log is maintained at the instrument to provide a record of which samples were run in each sequence and the internal standard areas for each run.

10.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Handling standard prep	Hazardous standards	Gloves and glasses required
Job Task #2:	**Hazards**	**Preventative Measures**
Loading samples	**Teledyne Tekmar Atomix autosampler might break a vial**	Glasses required
Job Task #3:	**Hazards**	**Preventative Measures**
GC maintenance	Electrocution	Unplug electrical outlets.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

TABLE 4
RECOMMENDED 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	0.100	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	0.100	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	0.100	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	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	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

TABLE 4
RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
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

920.0 NW-TPH Gas/ EPA-8021 BTEX
Soil, Water, and Air

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920.0 NW TPH GAS/ EPA-8021 BTEX

SOIL, WATER, AND AIR

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QA Manager – Glen Perry

Approved By: ____________________Rick Bagan_______ Date: ______8/13/2018__
Laboratory Director – Rick Bagan

Archival Date:	___________	Doc Control ID#:	____________	Editor:	____________

Analysis of MTBE, BTEX and Volatile Range Products by GC

1.0 Purpose

1.1 To define the procedure used to analyze for presence of volatile petroleum products, MTBE, and BTEX in environmental water, soil and air samples.

2.0 References

2.1 ALSEV Quality Assurance Manual (QAM).

2.2 EPA SW-846, Method 8021B.

2.3 "NWTPH-Gx - Volatile Petroleum Products Method for Soil and Water Analyses", Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology.

2.4 Hewlett Packard ChemStation and EnviroQuant users manuals.

2.5 ALSEV NWTPH-Gx Reference Chromatogram Library.

3.0 Definitions

3.1 Analytical Batch - The basic unit for analytical quality control. An analytical batch represents samples that are analyzed together with the same method, the same reagent lots and the same steps, within the same time period or within one week. The maximum batch size is 20 samples.

3.2 Method Blank - 40mL of analyte free water spiked with a surrogate at 10ppb. This quality control sample undergoes the same preparation and analytical procedure as the rest of the analytical batch. It is used to assess whether reagents used in the analysis are contaminated with any of the analytes of interest.

3.3 Blank Spike - A method blank spiked with surrogate at 10ppb and spike compounds of target analytes at 20ppb for BTEX and 500ppb for gasoline. A blank spike is used to assess the effectiveness of the analytical technique in recovering the compounds of interest from a clean matrix. Blank spikes are done in duplicate to provide a measure of laboratory precision.

3.4 Matrix Spike/Matrix Spike Duplicate – A sample spiked as the blank spike is (see 3.3). These are only done if sufficient sample is provided.

3.5 High Level 5035A - A soil sample prepared using the 5035A guidelines for analysis of volatile organic compounds in a solid matrix. High level samples are preserved by methanol in the field.

3.6 Method Detection Limit (MDL) - A number (with units of concentration) generated according to the procedure described in 40 CFR, Part 136, Appendix B. Theoretically, the MDL is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.7 Reporting Limit - The smallest amount of analyte that can be detected and reliably quantified. The reporting limit is normally the LLQC.

3.8 Lower Limit of Quantitation (LLQC) – The lowest concentration analytes may be measured and reported, which also must be ≥ the lowest point on the calibration curve.

3.8.1 The LLQC must be verified annually on every instrument where analysis takes place or whenever a significant change to the instrument is made. Recovery limits must be within the specified range of LCS ± 20% of the known concentration.

4.0 Apparatus and Materials

4.1 Purge and trap GC FID/PID analytical system consisting of the following:

4.1.1 Aquatec70 Vial Autosampler.

4.1.2 Vial-based autosampler (Archon-type).

4.1.3 Teledyne Tekmar Velocity with Tekmar Purge Trap A, and Stratum XPT Purge and Trap Sample Concentrators with Tenax Trap #1A (OI 4560 series).

4.1.4 Hewlett-Packard 5890 Series II Gas Chromatograph (GC) with J&W 0.53mm DB-5 column (or equivalent).

4.1.5 Agilent 7890B Gas Chromatograph (GC) with Zebron 0.53mm I.D. 60m column with 1.50 um film thickness ZB-5 column (or equivalent).

4.1.6 OI Analytical 4410 Flame Ionization Detector.

4.1.7 OI Analytical 4430 Photoionization Detector.

4.1.8 IBM compatible PC running HP MS ChemStation and EnviroQuant software.

4.1.9 AtomX Autosampler/Concentrator purge and trap system for 7890B GC.

4.2 40mL VOA vials with caps and septa.

4.3 5-mL Luerlock glass syringe

4.4 Micro-syringes, gas-tight, various volumes from 5uL to 1000uL.

4.5 Analytical balance – 0.01g

5.0 Reagents and Standards

5.1 Purge and trap grade methanol – High purity methanol, certified to be free of volatile analytes (obtained commercially).

5.2 Surrogate solution – Solution containing the surrogate Triflourotoluene (TFT) at 100ppb prepared from commercially available stocks. Prepared in purge and trap grade methanol. Example preparation: 500uL of a 2000ng/mL stock solution added to 10mL methanol and brought to a final volume of 10mL.

5.3 BTEX/MtBE calibration solution – Solution containing Benzene, Toluene, Ethyl Benzene, Methyl tert-Butyl Ether, and m,p,o-Xylenes prepared from commercially available stocks. Prepared in purge and trap grade methanol. Example preparation and concentration: 100uL of a 2000ppb MtBE stock solution and 500uL of a

200ppb BTEX stock solution added to methanol and brought to a final volume of 10mL and a final concentration of 20ppb for MtBE and 10ppb for BTEX compounds.

5.4 Gasoline calibration solution – Solution containing a mixture of unleaded, leaded, and premium commercial gasoline at 100ppb prepared from commercially available stocks. Prepared in purge and trap grade methanol. Example preparation: 400uL of a 2500ng/mL stock solution added to methanol and brought to a final volume of 10mL. (see 7.2)

5.4.1 Similarly, other products may be analyzed and calibrated using the FID. These may include Napthalene, Kerosene, Mineral Spirits, Jet Fuel A, and Aviation Gas (see 6.3 and 7.7 for more information on air samples). These other products are prepared like gasoline. Instead of containing a mixture of gasoline products, the solution will have the product of choice inside. Here we also use purge and trap grade methanol.

5.5 BTEX/MtBE spike solution – Solution containing Benzene, Toluene, Ethyl Benzene, Methyl tert-Butyl Ether, and m,p,o-Xylenes prepared from commercially available stocks other than that used to prepare the BTEX/MtBE calibration solution. Prepared in purge and trap grade methanol. Example preparation and concentration: 100uL of a 2000ppb MtBE stock solution and 500uL of a 200ppb BtEX stock solution added to methanol and brought to a final volume of 10mL and a final concentration of 20ppb for MtBE and 10ppb for BTEX compounds.

6.0 Sample Collection, Preservation, and Storage

6.1 Water samples are collected in 40mL VOA vials with no headspace and preserved with HCl at pH<2.

6.2 Soil/solid samples are collected in 4oz. jars, packed tight to minimize headspace or collected by 5035A.

6.3 Air samples are collected in Tedlar bags.

6.4 Water samples and soil samples collected in jars are stored at 2 to 6C and must be analyzed within 14 days of collection. High level 5035A (methanol preserved) samples are stored at 2 to 6C and must be analyzed within 14 days of collection. Air samples are stored at room temperature and must be analyzed within 72 hours of collection.

7.0 **Procedure** – Normal daily operations consist of verifying continuing calibration, verifying instrument cleanliness, and analysis of samples. An initial calibration is performed only when necessary (see 8.2). Due to the complexity of the data system it is assumed that the operator has read and is familiar with the MS ChemStation and EnviroQuant manuals.

7.1 **Instrument Operation** – Systems will vary depending on manufacturer and model. The operator is assumed to be familiar with the automated sampling system and data acquisition software. The operator will load the required samples, set up the purge and trap system to automatically run, and then ensure that the instrument follows the method-specified purge and trap procedure.

7.1.1 The operator will spike the sample with 40uL of the 10ppb surrogate standard (see 5.2).

7.1.2 The spiked sample will then be loaded onto the Archon, Aquatek 70, and/or Atomx autosampler(s) and the operator will set up the purge and trap system to run (see Appendix I for purge and trap operating parameters).

7.1.3 When the purge and trap desorbs the sample onto the GC FID/PID the instrument will automatically start. The GC will ramp to a column-appropriate maximum temperature at a rate that will allow sufficient separation of target analytes to allow accurate quantitation as determined by the analyst.

7.2 **Initial Calibration**

7.2.1 For Archon and Teledyne Aquatek 70 the operator fills 40ml VOA vial with H2O.

7.2.2 An appropriate amount of the calibration solution is added into the 40ml VOA vial to bring the concentration of the target compounds to the required level (40uL of the BTEX/MtBE calibration solution solution would be added to create a 10ppb calibration level, for example).

7.2.3 The spiked VOA vial containing the calibration level is then loaded onto the Autosampler(s).

7.2.4 A typical calibration curve for BTEX/MTBE will have calibration levels of 1, 5, 10, 50, 100, and 200ug/L for Benzene, Toluene, Ethyl Benzene and o-Xylene (MtBE and m,p-Xylene levels would be 2, 10, 20, 100, 200, and 400ug/L, respectively).

7.2.5 A typical calibration curve for gasoline ranges will have calibration levels of 50, 100, 200, 500, 1000, and 2000ug/L. See Appendix II for a guide to integrating the various gasoline ranges.

7.2.6 Only two types of calibration curve may be used. Level 1, which is an average curve, or level 2, which is a linear curve. Both of these only need five-points and are therefore the only ones that may be used.

7.2.7 Calibration Update

7.2.7.1 Once all of the standards have been analyzed the quantitation report for each calibration level must be checked for errors. Some manual integration will be necessary as the automatic quantitation routines will miss some of the compounds in the low concentrations and incorrectly integrate some compounds in the high concentration standards.

7.2.7.2 The analyst then loads the calibration curve into the Chemstation software and verifies that it meets the calibration requirements as specified in 8021B:

7.2.7.2.1 At least 5 calibration levels exist for each target analyte. Calibration levels may be removed at the high end or the low end of the calibration for a target analyte provided that at least 5 levels remain and no levels are removed between the high and low end of the calibration range (no ‘holes’ in the calibration, if you will). The lowest calibration level should represent the equivalent of the reporting limit or be near (yet above) the MDL.

7.2.7.2.2 All target analytes must have a relative standard deviation (RSD) equal to or less than 20 if the average of the response factors is to be used as the curve fit.

7.2.7.2.3 All target analytes must have a coefficient of determination (r^2) equal to or greater than 0.995 if a linear regression is to be used as the curve fit.

7.2.8 Once the calibration update is complete a midpoint standard is prepared from stocks obtained from a source different from that used for the calibration. This standard is run and quantitated against the new initial calibration to verify the accuracy of the calibration. The calculated concentration of target analytes in the second source must match that of the initial calibration +/- 20%.

7.3 Retention Time Window

7.3.1 Retention time windows should be established by making three injections of a calibration standard within a 72-hour period. The standard deviation of the retention times found in these three injections is then calculated and the retention time window for each component is established as +/-3 standard deviations from the mean. It is important that the instrument is running within optimum operating conditions when retention time windows are established.

7.4 Continuing Calibration

7.4.1 Before samples can be analyzed the calibration must be verified by the analysis of a midpoint standard. This standard is injected at the beginning and the end of each analytical sequence and also after every 10 sample injections within the analytical sequence.

7.4.2 For sample data to be acceptable the continuing calibration standards bracketing an analytical sample must recover the target compounds within

+/-20% of the spiked value for BTEX compounds and +/-20% for Gasoline.

7.4.3 If the values obtained from a continuing calibration standard exceed +/-20% of the known value corrective action must be taken (see 8.2).

7.5 Method Blank

7.5.1 Once an acceptable continuing calibration is obtained a method blank is run to verify the cleanliness of the analytical system and reagents. Analysis of samples cannot begin until a method blank is run that recovers all target analytes below their respective reporting limits.

7.6 Quality Control Samples

7.6.1 A Blank Spike and Blank Spike Duplicate must be run for every analytical batch or every 7 days, whichever is more frequent.

7.6.1.1 Calculate the percent recovery in the BS and BSD then compare to the current criteria for this procedure. If the recovery meets the acceptance criteria, sample processing may proceed. If the recovery fails to meet criteria diagnose the problem and discuss it with the laboratory director or QC officer to determine what corrective action should be taken.

$$\% \text{ Recovery} = \frac{S_o}{A_c} \times 100$$

S_o - Observed Spiked Sample Concentration
A_c - Actual Spike Concentration

7.6.1.2 Calculate the relative percent difference (RPD) for duplicate analyses and compare to the current criteria for this procedure. If the RPD meets the acceptance criteria, sample processing may proceed. If the RPD fails to meet criteria, diagnose the problem, and discuss with the laboratory director or QC officer to determine what corrective action should be taken. Use the following equation where D_1 and D_2 represent results from duplicate analyses:

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

7.7 Analytical Samples

7.7.1 Water samples are run on the Archon or Aquatek70 autosamplers. VOA vial is spiked with 40ul of the 10ppb water surrogate, and placed in the autosampler tray.

7.7.2 Air samples may also be run using a soil method through the Archon autosampler. A 40mL VOA vial is then filled with 10mL of analyte free water spiked with 10uL of the 10ppb water surrogate standard and capped. Using an air tight 10mL gas syringe, 10mL of the air sample is then directly injected through the septum and into the vial and placed onto the Archon autosampler.

7.7.3 For high level 5035A samples and other soil samples a methanol extraction step is included before analysis.

7.7.3.1 High level 5035A sample vials are weighed and the sample weight is calculated. Other soil samples are prepared as follows: 5g of the sample is weighed into a 40mL VOA vial and 5mL of purge and trap grade methanol is added, and 25uL of the 100ppm surrogate standard. The sample/extract is agitated by placing it in a sonic bath for 3 minutes and centrifuged for 2 minutes.

7.7.3.2 Soil extracts are run on Archon or Aquatek 70 autosamplers, 800ul of the methanol extract is transferred to a VOA vial filled with 40ml analyte free water and placed onto the autosamplers.

7.7.3.3 The sample is then run just like a water sample. QC acceptance criteria for soils are used to evaluate the results.

7.7.3.4 After analysis the solvent volume must be adjusted based on the dry weight of the soil sample. Using the percent solids and the sample weight (as taken from the VOA vial), we can calculate the dry sample weight (g) and the volume (mL).

%solids*sample weight=**dry sample weight**

dry sample weigth-(sample weight +5)=**volume**
(we add 5 to the sample weight to account for the methanol that was added before analysis)

8.0 Quality Control

8.1 Performance Criteria

8.1.1 The limits for recovery of surrogates and spikes as well as the limits for RPD in duplicate samples are published in the "ALS Lims System"

8.2 Corrective Action

8.2.1 If in a continuing calibration the response of one or more target compounds is different from that of the initial calibration by more than 15% or 20% (depending on the compound in question) the calibration of the chromatographic system is suspect and the cause must be determined.

8.2.1.1 Check the autosampler purge position.

8.2.1.2 Troubleshoot between the detectors. If the PID response is decreasing the lamp may be failing. Increase the intensity on the PID controller and be prepared to replace the lamp. If the FID response is decreasing it is possible that the jet may be plugged or clogged (note that this is rather unlikely and other corrective action should be considered before the tricky maneuver of changing the jet is undertaken).

8.2.1.3 Shifting retention time windows and erratic response may be due to a rusting concentrator solenoid releasing improperly or the concentrator 6-port valve actuating improperly. Clean and check the concentrator.

8.2.1.4 Low response may also indicate a degraded or contaminated trap. Condition or replace the trap.

8.2.1.5 It is possible, although highly unlikely that the cause of the failing continuing calibration is any of the following:

- 8.2.1.5.1 Failing column (most GC columns will last upwards of 8 years with constant use).
- 8.2.1.5.2 Contaminated concentrator transfer line.
- 8.2.1.5.3 Contaminated purge lines in autosampler.
- 8.2.1.5.4 Active sites on water manager (if present) or other locations.

8.2.1.6 If the cause of the continuing calibration failure is not immediately apparent and there is no other reason to believe that the instrument is in need of maintenance simply run a new initial calibration.

8.2.2 If the surrogate recoveries in a sample analysis are outside of recovery limits the sample must be reanalyzed. If the surrogate(s) are still outside recovery limits it is considered to be due to the impact of the sample matrix and no further reanalysis is required.

8.2.3 If a sample has a result for one or more target compounds outside of the calibration range the sample must be diluted sufficiently and reanalyzed to bring the result(s) into linear range. .

9.0 Records Management

9.1 The calibration results are documented and filed in calibration files.

9.2 The preparation of standards is documented and filed in a bound notebook.

9.3 All blank and sample results are submitted in the appropriate project folder along with a summary of the relevant quality control data.

9.4 Continuing calibration standards, run sequences and other instrument data are filed in daily instrument files and stored on site in the company store room.

9.5 Analytical data is backed up on a monthly basis by the database administrator and stored on site.

10.0 Health and Safety Warnings

10.1 Each sample should be treated as a potential health hazard. Appropriate PPE must be worn, and safety procedures as prescribed in the Chemical Hygiene Plan must be observed.

11.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Handling samples and Gx/BTEX standards.	Chemical hazards; Methanol solvent, gasoline and BTEX standards, and samples themselves.	Familiarize oneself with the MSDS of all known chemicals being used as well as ones that the operator may come in contact with; wear proper PPE. (for particularly hazardous samples work under a hood)
Job Task #2:	**Hazards**	**Preventative Measures**
Working with glassware and syringes.	Physical damage will be sustained if a syringe needle or broken glassware cuts or punctures the skin.	Review the glassware and equipment safety sheet and take great care to avoid any sharp pieces of equipment broken or not; Know where the first aid kit is located.
Job Task #3:	**Hazards**	**Preventative Measures**
GC Maintenance	Electrocution hazard	Turn off targeted GC equipment before running maintenance; avoid loose wiring.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

Appendix I

Purge-and-Trap Operating Parameters

	Time (min)	Temperature (degrees C)
Purge	10	40
Desorb	2	180
Bake Out	10	180

Oven Settings

Carrier gas flow rate (He)	8mL/min
Temperature program	
Initial temperature:	40 C
Initial time:	6 min
Program:	8 C/min to 180 C
Final temperuature:	20 C/min to 220 C hold for 0.5 min
Injector temperature:	200 C
Detector temperature:	235 C

Appendix II

Gas Range Integration Parameters

- For gasoline or unidentified volatile range hydrocarbons, the area of the components from the start of toluene (C6) through dodecane (C12) is integrated to the baseline as a group. This includes resolved peaks and the underlying unresolved envelope that is typically seen in petroleum products.

- For mineral spirits, the area of the components from octane (C8) to dodecane (C12) is integrated to the baseline as a group. This includes resolved peaks and the underlying unresolved envelope that is typically seen in petroleum products.

- For JP4, the area of components from pentane (C5) to octane (C8) is integrated to the baseline as a group. This includes resolved peaks and the underlying unresolved envelope that is typically seen in petroleum products.

- For aviation gasoline, the area of components from propane (C3) to decane (C10) is integrated to the baseline as a group. This includes resolved peaks and the underlying unresolved envelope that is typically seen in petroleum products.

941.1 NWTPH DX WATER

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941.1 NWTPH DX WATER

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Approved By:	____________________Rick Bagan______ Laboratory Director – Rick Bagan	Date:	__________8/13/2018_

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Analysis of Semi-Volatile Petroleum Products in Water (NWTPH-Dx)

1.0 Purpose

1.1 To outline the procedure for the analysis of semivolatile petroleum products in water using gas chromatography. The base method is taken from Washington State Department of Ecology (see 2.2 below).

2.0 References

2.1 ALSEV Quality Assurance Manual (QAM).

2.2 Analytical Methods for Petroleum Hydrocarbons, NWTPH-Dx: Semivolatile Petroleum Products Method for Soil and Water, Washington State Department of Ecology.

2.3 ALSEV SOP# ALSEV 940, Analysis of Semivolatile Petroleum Products in Soil (NWTPH-Dx).

2.4 ALSEV NWTPH-Dx Reference Chromatogram Library.

3.0 Definitions

3.1 Semi-Volatile Petroleum Products - Hydrocarbons extracted with methylene chloride that have the majority of their components eluting outside of the gasoline range (>C12), i.e. jet fuels through heavy fuel oils.

3.2 Analytical Batch - The basic unit for analytical quality control. An analytical batch represents samples that are analyzed together with the same method, same lots of reagents, and same steps in common to each sample within the same time period or within one week. The maximum batch size is 20 samples.

3.3 Method Blank - An artificial sample designed to monitor the introduction of artifacts into the analytical scheme. The method blank is taken through each step of the analysis.

3.4 Continuing Calibration Standard (CCS) - A mid-range working standard used to verify that the instrument is functioning correctly and that the calibration is still valid. The value obtained for this analysis must not vary from the true value by more than ±15%. If the value falls outside of this range then a second mid-range standard should be analyzed. If the analysis of the second check standard fails to meet acceptance criteria, then corrective action must be taken prior to any sample analyses.

3.5 Surrogate - A surrogate is an organic compound that is similar to the analytes of interest in chemical composition, extraction and chromatographic properties, but which is not normally found in environmental samples. Surrogate compounds are spiked into all blanks, standards and samples before analysis. Percent recoveries are calculated for each surrogate. Suggested surrogates for this method include 2-fluorobiphenyl, o- or p-terphenyl or pentacosane.

3.6 Blank Spike (BS) - A quality control sample, which has been spiked with a second source spiking solution at a known concentration and prepared independently from the standard used for calibration. The BS is carried through the analysis. Results of the blank spike are used to monitor method performance and accuracy on an on-going basis, and must fall within acceptable limits in order for the accompanying sample in the analytical batch to be valid.

3.7 Blank Spike Duplicate (BSD) – A quality control sample which has been spiked with the same second source spiking solution and concentration that was used in preparing the BS sample. The BSD is carried through the analysis. Results of the BSD are used to monitor method accuracy and precision on an on-going basis, and must fall within acceptable limits in order for the accompanying samples in the analytical batch to be valid.

3.8 Method Detection Limit (MDL) - A number, with units of concentration, generated according to the procedure described in 40 CFR, Part 136, Appendix B. The MDL is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.9 Diesel calibration standard (DCS) - Equivalent hydrocarbon mixture in which greater than 95% of the hydrocarbon mass elutes within the diesel range diluted to the appropriate concentrations in methylene chloride.

3.10 Motor Oil Calibration Standard - Equivalent hydrocarbon mixture in which greater than 95% of the hydrocarbon mass elutes within the motor oil range (>C24) diluted to the appropriate concentrations in methylene chloride.

3.11 Reporting Limit - The smallest amount of analyte that can be detected and reliably quantified and is based on the lowest standard. For this method the reporting limit is 130 ug/L for petroleum products in the elution range of jet fuels through #2 diesel and 250 ug/L for petroleum products eluting after #2 diesel, e.g. motor oils, hydraulic fluids and heavy oils.

3.12 Second Source Calibration Standard – A calibration standard purchased or prepared from a source independent from the primary calibration standard. It is used in preparing the blank spike, blank spike duplicate and matrix spike samples to assist in verifying the accuracy of the initial calibration curve.

4.0 Apparatus and Materials

4.1 Analytical Instruments

4.1.1 Hewlett-Packard 6890/7890B series gas chromatograph (GC) flame ionization detector (FID) with temperature programmable oven, capillary inlet system and autosampler.

4.1.2 Hewlett-Packard Chemstation data system compatible with GC that is capable of integrating and summing total area responses.

4.1.3 Suggested GC column: 30 meter x 0.32 mm ID, DB-5 with 0.25 µm film thickness.

4.1.4 GC accessories including, but not limited to column supplies, syringes, vials and closures, compressed gases and filters, and septa.

4.2 Sample Preparation Equipment

4.2.1 Balances

4.2.1.1 Analytical balance, capable of weighing to 0.1 mg.

4.2.1.2 Top loading balance, capable of weighing to 0.01g.

4.2.2 Syringes (various volumes, from 10 to 1000 mL).

4.2.3 Separatory funnels, 1000-mL with Teflon stopcocks.

4.2.4 Kuderna-Danish (KD) 250-mL evaporating apparatus with 10-mL concentrator tube, 3-ball macro Snyder column and 2-ball micro Snyder column.

4.2.5 Boiling chips.

4.2.6 Filter paper (rinsed with methylene chloride)

4.2.7 Oven, standard laboratory-type.

4.2.8 Miscellaneous glassware typically used in an analytical laboratory such as funnels, pipettes, vials and closures, beakers, volumetric flasks, pH indicator strips, magnetic stirrers, stir bars and ultrasonic baths.

5.0 Reagents

5.1 Methylene Chloride, gas chromatographic grade or equivalent.

5.2 Sodium Sulfate, anhydrous, granular, methylene chloride rinsed and/or baked in a muffle furnace.

5.3 1:1 HCL (for adjusting pH); Concentrated Sulfuric Acid and Silica Gel (for sample cleanup)

5.4 Standards

5.4.1 Diesel Calibration Standard - The stock diesel standard is purchased from vendors. Store at 4° (±2°) C. The stock standard is replaced after one year or sooner, if comparison to check standard indicates >15% difference.

5.4.2 Motor Oil Calibration Standard - The stock motor oil standard is purchased from any retail store selling national brand non-synthetic SAE 30 weight motor oil. Store the neat solution at room temperature and any working standards at 4° (±2°) C. The stock standard is to be replaced after one year or sooner, if comparison to check standard indicates >15% difference.

5.4.2.1 Additional calibration standards, e.g. Jet-A, Bunker-C, automatic transmission fluid (ATF) and transformer oil may be obtained from retail stores or from companies who use the petroleum products in their pure form.

5.4.3 Stock Surrogate Standard - The surrogate used in this analysis is n-pentacosane (C-25). The neat material is purchased from a vendor and a stock standard is prepared at 2500 mg/L in methylene chloride (as per WA DOE Analytical Methods). Store at 4° (±2°) C. The stock standard is to be replaced after one year or sooner, if comparison to check standard indicates >15% difference.

5.4.4 Calibration standards - The calibration standards are prepared by diluting the stock standard and stock surrogate standard in methylene chloride. The calibration standards are prepared from 50 to 5000 mg/L for diesel, 100 to 2500 mg/L for motor oil and 2 to 100mg/L for surrogate. The stock standard is to be replaced after one year or sooner, if comparison to check standard indicates >15% difference.

6.0 Sample Collection, Preservation and Handling

6.1 Samples are normally collected in 0.5-L amber glass containers with Teflon lines closures.

6.2 Samples are shipped in coolers with coolant and appropriate packaging to prevent cross-contamination and breakage.

6.3 Samples are to be extracted within 7 days of collection time if not preserved in 1:1 HCl and 14 days from collection if preserved in 1:1 HCl.

6.4 If the samples were collected in a larger container thoroughly shake samples in order to collect a representative subsample prior to extraction.

7.0 Procedure

7.1 Calibration

7.1.1 Prepare calibration standards at a minimum of five levels to define the working range of the FID. The lowest standard should represent the equivalent to the reporting limit or be near, yet above the MDL. Calibration curves shall be constructed for diesel, motor oil, Jet A, transformer oil, ATF and bunker C.

7.1.2 For #2 diesel, the area of the components after dodecane (C-12) through tetracosane (C-24) is integrated to the baseline as a group. This includes resolved peaks and the underlying unresolved area (hump) that is typically seen in petroleum products.

7.1.3 For motor oil, the area of the components after tetracosane (C-24) to the end of C36 is integrated.

7.1.4 Use the data system to determine the response and linearity of the calibration standards. If the correlation coefficient is >0.995, the calibration curve is assumed linear.

7.1.5 The initial calibration curve is further validated by analyzing a mid level second source diesel standard. The result should be within 15% of the standard concentration

7.2 Sample Extraction

7.2.1 Mark the meniscus of the sample bottle for later use in volume determination. Pour out the entire sample into a 1-L separatory funnel.

7.2.1.1 For samples with soil sediment in the bottle measure the meniscus of the sample bottle as well as the level of the sediment and carefully pour out the water sample into a 1-L separatory funnel so as not to disturb the sediment.

7.2.2 Adjust the pH of water sample to approximately 2 with the addition of 1:1 HCL and note the pH in the Extraction Log Book.

7.2.3 Add 16 µL of the surrogate working standard to the MB, BS, BSD and the field samples. Add 25 µL of the spike solution to the QC samples (BS,BSD).

7.2.3.1 The analytical batch consists of 20 samples. The following quality assurance samples must be analyzed with each batch or each day whichever is sooner:

1 **method blank**

1 **blank spike**

1 **blank spike duplicate**

7.2.3.2 Use 400 mL of DI water for each QC sample to be analyzed.

7.2.4 Add 30 ml of methylene chloride to the sample bottle and rotate the bottle at a sufficient angle to wash the walls. Pour the solvent into the separatory funnel containing the water sample.

7.2.4.1 For samples with sediment washing the sample bottle with the solvent is not done. Add 30 mL of methylene chloride directly into the separatory funnel.
7.4.2.1.2 To maintain the original solvent/sample ratio increase the quantity of the solvent for larger sample volume.

7.2.5 Place the Teflon cap on the separatory funnel and invert the funnel, making sure to open the stopcock with the stopcock end raised (venting). Shake vigorously several times while venting frequently.

7.2.6 Once the excess pressure has been vented, shake the separatory funnel vigorously for 1 minute.

7.2.7 Allow the two phases to separate, and then drain the solvent layer into a dry 250-mL Erlenmeyer flask.

7.2.8 Add 30 mL of methylene chloride and repeat steps 7.2.5 through 7.2.7.

7.2.9 Repeat 7.2.8 to complete a total of three one-minute shakes per sample.

7.2.10 Transfer the solvent extract through a glass funnel lined with filter paper and filled with sodium sulfate into a 250-mL KD flask attached to a 10-mL concentrator tube, rinsing out the Erlenmeyer flask with methylene chloride.

7.2.11 Rinse the sodium sulfate and the filter paper with 30 mL of methylene chloride and allow to drain.

7.2.12 Add a boiling stone to the liquid and attach a 3 ball macro Snyder column. Concentrate extract to approximately 5 mL. Allow to cool.

7.2.13 Remove 3-ball macro Snyder column and KD flask from the concentrator tube. Add a new boiling stone into the concentrator tube and place a 2-ball micro Snyder column on the tube.

7.2.14 Concentrate extract to less than 1.0 mL on the steam bath then remove and allow to cool.

7.2.15 Bring extract to 1 mL final volume with methylene chloride using a 1-mL syringe.

7.2.16 Prepare 2 of the 2-mL autosampler vials for GC analysis, placing 500µL into each vial. Label the vial with the laboratory sample ID and extraction procedure. Store in a refrigerator at 4° (± 2°) C until analysis.

7.3 Sample Extraction for Low Level Reporting/Analysis

Follow the extraction procedure as described in 7.2 except for the following:

7.3.1 For the QC samples (MB, BS, BSD) use 1000 mL DI water.

7.3.2 Use 50 mL methylene chloride for extracting.

7.3.3 Shake the samples for 2 minutes on the first, 2 minutes on the second and 1 minute on the third shake.

7.4 Sample Extraction for Decane and Octadecane Analysis

Follow the extraction procedure as described in 7.2. except for the following:

7.4.1 Use 32 µL of Pentacosane surrogate and spike QC samples (BS, BSD) with 25 µL of Diesel Range Organics Mix.

7.4.2 Bring the extract to a final volume of 10 mL with methylene chloride.

7.5 Sample Cleanup

If a sample contains a significant amount of naturally occurring non-petroleum organics which may contribute to biogenic interference or if the client has requested sample cleanup then the following procedure is performed.

7.5.1 Bring the extract to 1 mL final volume with methylene chloride using a 1-mL syringe and transfer it to a 2-mL autosampler vial.

7.5.2 Using a disposable pipet, add a small drop of concentrated sulfuric acid.

7.4.3 Cap the vial and shake for about 30 seconds. Centrifuge to separate 2 phases.

7.5.4 Using a disposable pipet, carefully transfer the methylene chloride (top) phase into another autosampler vial and add a small amount (about a pinch) of silica gel to the extract.

7.5.5 Cap the vial and shake for about 30 seconds. Centriguge to separate 2 phases.

7.5.6 Using a disposable pipet, carefully transfer the methylene chloride (top) phase into a 2-mL autosampler vial for GC analysis. Label the vial with Lab ID and extraction/cleanup procedure. Store in a refrigerator at 4° (± 2°) C until analysis.

7.6 Sample Analysis

7.6.1 Gas Chromatograph Analysis

7.6.1.1 Samples are analyzed by GC/FID. Optimum injection volume of 1 µL is recommended.

7.6.1.2 If initial calibration has been performed, verify the calibration by the analysis of a midpoint CCS for diesel and motor oil. The standard is injected at the beginning and at the end of the analytical analytical sequence, as well as after 10 sample injections within the analytical analysis.

7.6.1.2.1 Diesel #2 and motor oil shall be used as the default petroleum products for reporting purposes when no petroleum products were identified in any initial screening or when the types of petroleum products are unknown prior to analysis.

7.6.1.2.2 CCS are analyzed for diesel and motor oil only. If the CCS is acceptable for diesel and motor oil, it is then assumed that the initial calibration curves for the other petroleum hydrocarbons are within the acceptable limits as well.

7.6.1.2.3 Periodically the validity of the initial calibration curves for the other products should be verified by the analysis of mid range standard.

7.6.1.3 Compare the result of the analyzed CCS with the true value. If the result has a percent difference greater than 15%, corrective action must be taken.

7.6.1.4 A solvent blank (methylene chloride) must be analyzed each day to determine the area generated from normal baseline noise under conditions prevailing in the 24 hour period.

7.6.1.4.1 Blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination above the reporting limits, subsequent blanks are analyzed until the system is shown to retain contaminate at concentrations less than the limits.

7.6.1.5 If the petroleum product concentration exceeds the linear range of the method (as defined by the range of the calibration curve) in the final extract, dilution or other corrective action must be taken. When analyzing a dilution it is best if the response of the major peaks is kept in the upper half of the linear range of the calibration curve.

7.6.1.6 Once the sample chromatograms have been generated, the observed petroleum product shall be determined by pattern matching with standard chromatograms referenced in section 2.4 or with current fingerprints that have been run by the laboratory.

7.6.1.6.1 If the chromatogram matches a reference chromatogram for a specific product the sample contaminant is identified as such.

7.6.1.6.2 If specific product identification cannot be made, quantitate the sample with the calibration curve for the petroleum product that most closely resembles that of the sample. In such cases, the sample product is identified as “product which is similar to . . .”.

7.6.1.6.3 The term “unidentified diesel range product” is used when specific identification is not possible for the petroleum products present that have an unresolved envelope that ends before tetracosane (C-24).

7.6.1.6.4 The term “unidentified lube oil product” is used when specific identification is not possible for unresolved chromatographic envelopes originating at, or extending beyond tetracosane.

7.6.1.6.5 For samples containing both diesel and motor oil products, integration points are adjusted in order to incorporate the majority of the components of the petroleum products identified as present in the sample.

7.6.1.6.6 If there is an overlap within the volatile and diesel ranges or within late diesel and early motor oil ranges, indicate on the report that the corresponding ranges are biased high due to product overlap.

7.6.1.6.7 For Decane (C10) and Dodecane (C18) analysis overlay comparison of the C10 and C18 peaks between the sample and the DRO standard (CCV) is performed.

7.6.2 Calculations

7.6.2.1 The data system calculates and prints the solution concentration for the sample extract. The analyst uses the solution concentration to calculate the sample result. The example calculation is:

Sample Results(ug/L) = (A x B) / C x 1000

Where:
A = Solution concentration (ug/L)
B = Final extract volume (mL)
C = Amount of water extracted (mL)

8.0 Quality Control

8.1 On-going quality control

8.1.1 Quality Control acceptance criteria are given in the ALSEV Control Limits Table.

8.1.2 Extract a method blank per section 7.2.3.1

8.1.2.1 The method blank must show a non-detect for petroleum products and is recorded as diesel #2 < 130 ug/L and motor oil <250 ug/L. If the method blank meets these acceptance criteria, then the integration may proceed.

8.1.3 Analyze the method blank sample for the analytical batch prior to the duplicates and field samples.

8.1.4 Extract a blank spike and a blank duplicate per section 7.2.3.1.

8.1.5 Calculate surrogate recovery for each QC sample and field sample and compare to the current acceptance criteria for this procedure. If the recovery meets the acceptance criteria, then sample results are acceptable. If the recovery fails to meet criteria, diagnose the problem and if necessary, repeat the sample extraction. The percent recovery is calculated as:

Surrogate % recovery = (Observed conc. / True conc.) x 100

8.1.6 Calculate the relative percent difference (RPD) for duplicate analyses using the following equation, where Dl and D2 represent the results from duplicate analyses:

RPD=D1-D2/(D1+D2)/2 x 100

Compare the RPD with the current acceptance criteria for this procedure. If the RPD meets the acceptance criteria and other batch QC samples are acceptable, all samples in the analytical batch are acceptable. If the RPD fails to meet criteria, diagnose the problem and discuss with the laboratory director or QC Officer to determine if the analytical batch is to be reported.

8.1.7 Extract a matrix spike if sufficient sample is provided by the client.

8.1.8 Calculate the percent recovery of the spike. Compare the percent recovery with the current acceptance criteria for this procedure. If the percent recovery meets the acceptance criteria, all samples in the analytical batch are acceptable. If the percent recovery fails to meet criteria, diagnose the problem and discuss with the laboratory director or QA officer to determine if the analytical batch is to be reported.

8.1.9 Method Detection Limit Determination

8.1.9.1 A method detection limit determination is performed using the procedure described in 40 CFR, Part 36, Appendix B.

8.1.9.2 The method detection limit determination is performed at least once to demonstrate confidence levels. Project specific plans may require additional determinations at specified frequencies.

8.2 Nonconformance and Corrective Action

8.2.1 Any discrepancy affecting the quality of the data for any sample is documented on a nonconformance memo (NCM) or within the project file.

9.0 Records Management

9.1 The analysis printout for the sample data is filed in the client project file. The analysis printout for the continuing calibration standards are filed in the instrument sequence files. Copies of the QC summary sheet and analysis printout for QC samples are filed with sample data in the project file. The analysis printout for initial calibration standards is filed in the calibration files.

9.2 The sample preparation information is entered into a bound notebook. The information is not routinely copied to the client file.

9.3 The preparation of standards is documented and filed in the standards file

10.0 Health and Safety Warnings

10.1 Each sample should be treated as a potential health hazard. Appropriate PPE must be worn and safety procedures in the Chemical Hygiene Plan must be observed.

SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Using solvent (Methylene chloride) and adding surrogate (Pentacosane) during extraction	Accidental spills and splashes	Use PPE (gloves, protective clothing, eye protection). Perform task under fumehood.
Job Task #2:	**Hazards**	**Preventative Measures**
Venting when shaking water samples	Inhalation of fumes	Perform task under fumehood.
Job Task #3:	**Hazards**	**Preventative Measures**
Using hot water bath to boil down extract	Inhalation of fumes	Perform task under fumehood. Place sash window down to the maximum protection level.
Job Task #4:	**Hazards**	**Preventative Measures**
Washing and handling glasswares	Skin cuts	Use PPE. Avoid using chipped/slightly broken glasswares.
Job Task #5:	**Hazards**	**Preventative Measures**
Disposal of excess or refuse water samples	Inhalation of fumes. Skin contact (from acids used as preservatives)	Use Sodium carbonate to neutralize water samples under fumehood, then pour out in sink. Use cold water to flash.
Job Task #6:	**Hazards**	**Preventative Measures**
Using Hydrocholric acid and silica gel to clean up extract	Skin contact	Use PPE.
Job Task #7:	**Hazards**	**Preventative Measures**

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

Method NWTPH-Dx, Appendix 1
Acceptance Criteria for Quality Control

	% Recovery	Relative % Difference
Continuing Calibration	85-115	
Surrogate Recovery	60-126	
Spike Duplicates	67-125.2	10.8

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY
REFERENCED METHOD:	SW8260C AND EPA 624
SOP ID:	525
REV. NUMBER:	18
EFFECTIVE DATE:	AUGUST 10, 2018

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 525 REVISION 18

TITLE: DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY -- METHODS SW8260C, or EPA 624

FORMS: NONE (instrument printout used as run log)

APPROVED BY:

PRIMARY AUTHOR__________________________________DATE _____________

QUALITY ASSURANCE MANAGER ________________________ DATE _____________

LABORATORY MANAGER __________________________________DATE _____________

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- SW-846 methods 5030C, 5035A and 8260C; also EPA 624 -- are used to determine volatile organic compounds in a variety of matrices. This SOP is applicable to nearly all types of samples, regardless of water content, including: groundwater, aqueous sludges, caustic or acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons or catalysts, soils, and sediments. The following compounds are presently being analyzed using this SOP. Other compounds can be analyzed after successful demonstration of capability (DOC) and method detection limits study (MDL). Analytes in the Table below are listed in typical elution order. Analytes that are part of ALS's standard reporting list are depicted in bold.

Parameter	CAS No[b]	Purge & Trap
dichlorodifluoromethane	75-71-8	A
chloromethane	74-87-3	A
vinyl chloride	75-01-4	A
bromomethane	74-83-9	A
chloroethane	75-00-3	A
trichlorofluoromethane	75-69-4	A
acrolein	107-02-8	A
1,1-dichloroethene	75-35-4	A
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	A
acetone	67-64-1	PP
iodomethane	74-88-4	A
carbon disulfide	75-15-0	PP

methylene chloride	75-09-2	A
trans-1,2-dichloroethene	156-60-5	A
methyl tertiary butyl ether	1634-04-4	A
acrylonitrile	107-13-1	A
1,1-dichloroethane	75-34-3	A
vinyl acetate	108-05-4	A
cis-1,2-dichloroethene	156-59-2	A
2-butanone	78-93-3	PP
bromochloromethane	74-97-5	A
chloroform	67-66-3	A
1,1,1-trichloroethane	71-55-6	A
2,2-dichloropropane	594-20-7	A
carbon tetrachloride	56-23-5	A
1,1-dichloropropene	563-58-6	A
1,2-dichloroethane	107-06-2	A
benzene	71-43-2	A
trichloroethene	79-01-6	A
1,2-dichloropropane	78-87-5	A
dibromomethane	74-95-3	A
bromodichloromethane	75-27-4	A
2-chloroethyl vinyl ether	110-75-8	A
cis-1,3-dichloropropene	10061-01-5	A
4-methyl-2-pentanone	108-10-1	PP
toluene	108-88-3	A
trans-1,3-dichloropropene	10061-02-6	A
1,1,2-trichloroethane	79-00-5	A
2-hexanone	591-78-6	PP
tetrachloroethene	127-18-4	A
1,3-dichloropropane	142-28-9	A
dibromochloromethane	124-48-1	A
1,2-dibromoethane	106-93-4	A
1-chlorohexane	544-10-5	A
chlorobenzene	108-90-7	A
1,1,1,2-tetrachloroethane	630-20-6	A
ethylbenzene	100-41-4	A
m- and p-xylene	108-38-3/106-42-3	A
o-xylene	95-47-6	A
styrene	100-42-5	A
bromoform	75-25-2	A

isopropylbenzene	98-82-8	A
1,2,3-trichloropropane	96-18-4	A
1,1,2,2-tetrachloroethane	79-34-5	A
bromobenzene	108-86-1	A
n-propylbenzene	103-65-1	A
2-chlorotoluene	95-49-8	A
1,3,5-trimethylbenzene	108-67-8	A
4-chlorotoluene	106-43-4	A
tert-butylbenzene	98-06-6	A
1,2,4-trimethylbenzene	95-63-6	A
sec-butylbenzene	135-98-8	A
1,3-dichlorobenzene	541-73-1	A
p-isopropyltoluene	99-87-6	A
1,4-dichlorobenzene	106-46-7	A
n-butylbenzene	104-51-8	A
1,2-dichlorobenzene	95-50-1	A
1,2-dibromo-3-chloropropane	96-12-8	PP
1,2,4-trichlorobenzene	120-82-1	A
hexachlorobutadiene	87-68-3	A
naphthalene	91-20-3	A
1,2,3-trichlorobenzene	87-61-6	A
trans-1,4-dichloro-2-butene	110-57-6	PP
acetonitrile	75-05-8	PP
allyl chloride	107-05-1	A
chloroprene	126-99-8	A
1,4-dioxane	123-91-1	PP
ethanol	64-17-5	PP
ethyl methacrylate	97-63-2	A
ethyl-tert-butyl ether	637-92-3	n/a
hexachloroethane	67-72-1	PP
isobutyl alcohol	78-83-1	PP
isopropyl ether	108-20-3	n/a
methacrylonitrile	126-98-7	PP
methyl methacrylate	80-62-6	A
propionitrile	107-12-0	PP
tert-amyl methyl ether	994-05-8	n/a
tert-butanol	75-65-0	n/a

A	Adequate response by this technique.
b	Chemical Abstract Services Registry Number.
PP	Poor purging efficiency resulting in high EQLs.
n/a	Not applicable; not designated in method.

This SOP describes purge & trap GC/MS procedures that can be used to identify and quantify most organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. However, for the more soluble compounds, quantification limits are approximately five to ten times higher because of poor purging efficiency. Ketones, alcohols and aldehydes are typical of classes of compounds that may have elevated reporting limits due to their high degree of water solubility.

Note that the body of this SOP specifies the procedures to be used for Methods SW8260 C. Any additional or contradictory requirements for EPA Method 624 are addressed in Section 10, and are compliant with the requirements of 40 CFR Part 136.6 as stated in the 2012 Method Update Rule (MUR).

When requested, samples may be analyzed for Gasoline Range Organics (GRO). The carbon range integrated for GRO extends from C6 to C10, which is identified by analyzing a gasoline component standard. A gasoline composite standard is used for initial calibration and the quantification of sample results. The concentration of GRO is calculated using the external standard technique, and the sum of all peak responses within the 2-methyl pentane to 1,2,4-trimethyl benzene retention time range.

2. SUMMARY

Volatile compounds are introduced into the gas chromatograph (GC) by purge & trap. Purged sample components are trapped in a tube containing suitable sorbents in accord with Methods SW5030C or SW5035A. When purging is complete, the sorbent tube is heated rapidly and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph.

As analytes elute from the capillary column, they are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantification) ion relative to an internal standard with the response factor or calibration equation generated from a multi-point calibration curve using average response factors or regression equations.

3. RESPONSIBILITIES

3.1 It is the responsibility of the Analyst to perform the analyses according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include

Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The ALS Project Manager is responsible for directing a chlorine residual check to be performed upon sample receipt as applicable.

3.5 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.6 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

3.7 If the words “QSM Criteria” appear on the WIP and Tracking Sheets for a specific work order, that work order requires the criteria as specified in Appendix B and C of SOP 996, or as defined in the appropriate Program Specification.

4. INTERFERENCES

4.1 **Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks and the above sources are suspected, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter and/or sorbent trap. Many trace impurities in the purge (carrier) gas are removed**

by passing the He through a heated catalyst bed that is capable of removing hydrocarbons and oxygen.

4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing the purge needle or apparatus and sample syringes with three portions of organic-free reagent water between samples. Sample tubes are only reused if washed and baked before the next use. After analysis of a sample containing high concentrations of volatile organic compounds, one or more reagent blanks should be analyzed to check for cross-contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purge needle or apparatus with methanol and then rinse it thoroughly with organic-free reagent water. In extreme situations, the entire sample pathway of the purge & trap may require dismantling and cleaning or replacement. The relatively low purging efficiency of many analytes from a large volume sample (e.g., 10mL, 25mL) often results in significant concentrations remaining in the sample purge tube after analysis. Archon autosamplers (or equivalent) use the same purge vessel repetitively for water analysis, but rinse the purge vessel with He and water between samples. If carryover contamination is suspected, (this is likely when a sample containing high concentration levels of volatile compounds is followed by a sample containing low levels of the same volatile compounds), all samples that may have been affected must be re-analyzed. Sample analysis may continue if a cleanup blank or sample following the high concentration sample is free (below the reporting limit) from compounds present over the calibration range in the high level sample. Analyst experience should be used to determine which compounds tend to carryover and at what levels.

4.2.1 Annotations made to instrument run logs should indicate if a sample contains possible carryover contamination. If the subsequent rerun of the sample confirms the presence and level of the volatile compounds, either analysis may be used. If, however, the rerun shows that the presence of the compounds was carryover contamination, only the rerun should be used. The original analysis should be considered non-usable data for the analytes that may have carried over.

4.3 Special precautions must be taken to analyze for methylene chloride. The GCMS Volatiles laboratory is located on the opposite side of the building from the Organic Extractions lab, in order to minimize the level of methylene chloride contamination. Because methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean because clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol serves as a check on such contamination. To check for cross-contamination during sample storage, the laboratory periodically analyzes sample storage refrigerator blanks (SOP 512).

5. EQUIPMENT AND SUPPLIES

5.1 PURGE & TRAP AUTOSAMPLER DEVICE

- Autosampler - OI 4552/Archon, Varian Archon, or equivalent
- Sample concentrator - OI 4560 Liquid Sample Concentrator equipped with OI #10 adsorbent trap, or equivalent.
- Autosampler/concentrator – Teledyne Tekmar Atomx Purge and Trap System with K trap or equivalent.

5.2 GAS CHROMATOGRAPH (GC), DETECTOR AND MASS SPECTRAL LIBRARY

Hewlett Packard (HP) Model 5890A or 6890 GC (or equivalent) capable of splitless or split/splitless injection or direct interface to a purge & trap apparatus. Equipped with variable constant differential flow controllers (so that the column flow rate will remain constant throughout desorption) and a temperature-programmable oven. Also equipped with a HP5971, 5972 or 5973 mass spectrometer detector (or equivalent), capable of scanning from 35 to 270amu every 1sec 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 p-bromofluoro-benzene (BFB) which meets all of the criteria in Table 1 (shown subsequently) when 50ng or less of the GC/MS tune standard is introduced through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC. The NBS/EPA/NIST mass spectral library (library may vary with instrument) is also used to identify non-target compounds generally known as tentatively identified compounds (TICs).

GC/MS interface to the mass spectrometer: Direct coupling by inserting the column into the mass spectrometer is generally used for 0.18 to 0.32mm-ID columns. Any enrichment device or transfer line can be used if all of the performance specifications described in this SOP (including tuning) can be achieved.

5.3 DATA ACQUISITION AND PROCESSING SYSTEM

A computer system that facilitates 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 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 specified time or scan-number limits.

5.4 COLUMNS - Equivalent columns/guard columns may also be used

Column 1 - 60m x 0.25mm ID capillary column with RTX-624 stationary phase (Restek), 1.4µm film thickness

Column 2 - 60m x 0.25mm ID capillary column with RTX-VMS (Restek), 1.4µm film thickness

5.5 GASES- **only high purity or higher grade gases may be used!**

- Helium: purge & trap and carrier gas

5.6 MEASURING DEVICES

- Microsyringes - 5, 10, 25, 50, 100, 250, 500, and 1,000µL
- Syringes - 5, 10, or 30mL, glass
- Syringe valve, two-way with Luer ends (three each), if applicable to the purging device
- Laboratory balance, 0.01g sensitivity (used for weighing solid samples); operated per SOP 305 requirements.

5.7 CONSUMABLE SUPPLIES

- Compact Vespel/Graphite Ferrule, Restek #20264 or equivalent
- Graphite Ferrules, various sizes
- Glass scintillation vials, 20mL and 40mL, with TeflonTM/-lined/low-level siloxane screw-caps, or, glass culture tubes with TeflonTM-lined screw-caps
- Vials, 2mL, with TeflonTM-lined screw-caps
- Pasteur pipettes, 5 ¾” and 5mL, disposable
- Volumetric pipettes, 10mL, Class A, disposable
- Volumetric flasks, Class A - 5mL, 50mL, and 100mL, with ground-glass stoppers

- Spatula, stainless steel
- pH paper, acidic narrow range and wide range
- PTFE-coated magnetic stir bars, for use in soils purged with the Archon autosamplers (SW5035, SW5035A)
- MininertTM or CERTANTM vials or equivalent

6. REAGENTS AND STANDARDS

6.1 **Organic-free reagent water (SOP 511)**

6.2 Methanol (CH_3OH), purge & trap quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents. J.T. Baker #907702 or equivalent

6.3 Pre-conditioned Ottawa sand (for use as clean matrix for method blank (MB) and laboratory control sample (LCS) analyses associated with solid matrix sample analyses). Pre-condition by drying in an oven set at 105°C or greater overnight; EMD #SX0075-3 or equivalent

6.4 STANDARDS

NOTE: Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards in methanol be stored at –10°C to –20°C in MininertTM or CERTANTM vials with TeflonTM-lined screw-caps. Stock standards that are not accessed as part of routine operations may be stored in 2mL glass vials with TeflonTM-lined caps (i.e., MininertTM vials are not required for rarely utilized stock standards).

6.4.1 All standards are maintained per SOP 300. Two independent sources of commercial target analyte stock standards, in methanol, are required. The stock standards are purchased as certified solutions from suitable vendors. Typically, concentrations of stock solutions vary from 1,000-10,000µg/mL.

6.4.2 Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampoules, if recommended by the manufacturer. Standards for this procedure must be equilibrated to —<0°C (stored in freezer) before opening and protected from light. After opening/initial use, transfer remaining stock standard to a suitable vial (CERTANTM vial with a TeflonTM-lined screw-cap) with minimal headspace, and store in a freezer (-—<0°C).

6.4.3 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds 20% drift. Standards for gases may need to be replaced after one week unless the acceptability of the standard can be documented.

Standards for the non-gases should also be monitored closely by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases may need to be replaced after one month for working standards and three months for opened stocks, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether or styrene may need to be prepared more frequently

NOTE: For initial calibrations, either the first or second source should be less than one month old for non-gas working standards (and 3 months for stocks) and one week for permanent gases.

6.4.4 First source materials are used to create calibration and continuing calibration verification (CCV) standards. Second source materials are used to create the initial calibration verification (ICV) solution. Laboratory control and matrix spike standards may be from either source.

Non-target analyte internal standard (IS) and surrogate (SS) stock standards are also purchased. The IS is used to quantitate analytes detected in samples. The SS is used to monitor system performance and method effectiveness with each sample matrix. The internal standards (IS) currently utilized for this method are: Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. The surrogates currently utilized are: Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8, 4-Bromofluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Other compounds may be used as surrogates, depending upon the analysis and client requirements. It is recommended that internal standards and surrogates be combined (intermediate solution) and prepared at a concentration of 50ug/mL (5uL injected) for the Atomx autosampler and 250ug/mL (1uL injected) for the Archon style autosampler. Each standard, sample or QC sample must me spiked with internal standards and surrogates prior to analysis.

NOTE: The surrogates may be spiked in the initial calibration standards at the same concentration as they are spiked in the samples themselves. Response factors for the surrogates are then averaged to produce a one-point calibration with the sole purpose of measuring the surrogate recovery using the same concentration for each sample analysis. Alternatively, the surrogates can be calibrated in the same manner as the targets themselves (i.e. varying concentrations). If this latter option is used, an equipment validation study must be performed to determine the actual volume of standard delivered. The concentration of standard may be adjusted accordingly for the actual volume delivered at the 1µL setting. For example: (1.135µL actual delivery)(441µg/mL IS/SS spiking solution)/5mL = 100µg/L.

Prepare intermediate QC spike standards, in methanol, from volatile organic compounds that will be representative of the compounds being investigated. At a minimum, the matrix spike will include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Consult applicable LIMS program specifications for appropriate compound list. See Section 9 of this SOP for further details regarding QC (i.e., LCS/LCSD, MS/MSD) samples.

6.4.5 4-bromofluorobenzene (BFB) tune standard: A standard solution containing 50ng/µL of BFB in methanol is prepared.

6.4.6 An appropriate volume of target analyte stock standard is diluted, with methanol, to a specific volume to create intermediate standards. The intermediate standard may contain the compounds of interest singly or mixed together. Intermediate standards must be stored with minimal headspace and should be checked frequently for signs of degradation, especially just prior to preparing working calibration standards from them. Store standards in an appropriate vial with minimal headspace. The standards may be retained as prescribed in SOP 300. All dilutions should be performed using syringes, and purge & trap grade MeOH.

6.4.7 Target analyte calibration (working) standards at a minimum of five concentrations should be prepared from the intermediate standards. Prepare these solutions in organic-free reagent water. One of the concentrations should be at a concentration less than or equal to the reporting limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. The laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s). Aqueous calibration (working) standards must be prepared on the day of loading on the autosampler.

To prepare a target analyte calibration standard for purge & trap, add an appropriate volume of an intermediate standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a micro syringe and rapidly inject the standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the capped flask three times only. Transfer the working standard to a 40mL VOC vial without headspace for low-level water analysis or 5mL into a 40mL VOC vial for soil analysis. It is also acceptable to add the appropriate amount of intermediate standard directly to a gas tight syringe containing the desired purge volume of organic-free water for a 5mL working standard. Archon autosamplers (or equivalent) add the internal standards and surrogates to the working calibration solution prior to analysis. Perform purge and trap procedures as outlined in Methods SW5030C or SE5035A.

6.4.8 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory department.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 **Samples should be collected according to an approved sampling plan.**

7.2 Samples from chlorinated water sources should be dechlorinated with sodium thiosulfate (Na2S2O3) in the field at the time of collection. These samples should then be acidified with hydrochloric acid (HCl) following dechlorination. Based upon project knowledge provided by the client, where applicable, ALS's Project Manager may instruct the volatiles analysts to test for chlorine residual just prior to preparation for analysis. A chlorine residual test kit, obtainable from the Sample Receiving Department, is used to check for chlorine residual. Notify the Project Manager immediately if residual chlorine is present.

7.3 Volatile organic analysis of water and soil samples extracted by Methods SW5030C or SW5035A must be performed within 14 days of collection unless otherwise specified by the client. Water samples are usually preserved by adding approximately four (4) drops of concentrated hydrochloric acid (HCl) to each 40mL VOA vial. The purpose of the hydrochloric acid is to prevent microbial degradation of target compounds. If the water sample is unpreserved, the holding time may be shortened to seven (7) days from the date of collection. Volatile organic analysis of soil samples received in EnCore™ (or equivalent) samplers to be extracted by Method SW5035A shall be frozen upon receipt and analyzed within 14 days of collection. Other types of collection and preservation techniques may be required by Method SW5035A and should be evaluated according to the specific needs of the client. Other means of preservation for samples to be prepared for analysis by Method SW5035A include freezing soil in a 40mL vial after addition of water and a stir bar, as well as addition of sodium bisulfate solution ($NaHSO_4$) and a stir bar. Method SW5035A also allows preservation with methanol for solid samples with expected higher concentrations of target analytes. Preservation of samples for subsequent analysis via Methods SW5035A/8260C may be required within 48hrs after time of collection. Consult applicable LIMS program specification.

7.4 Following sample analysis, measure the pH of each sample. Record the result next to the sample's identity on the previously prepared daily sequence log. If the pH of a preserved sample is >2, immediately notify the appropriate Project Manager and discuss the pH excursion in the data package case narrative. Aqueous samples that are intentionally not preserved at the time of collection do not require Project Management notification.

7.5 Samples to be prepared by Method SW5030C must be collected in glass containers with minimal headspace and stored at 4±2°C. Samples to be prepared by Method SW5035A should be collected in EnCore™ (or equivalent) sampling devices and stored at <-7°C, but no less than -20°C. Other types of collection and preservation techniques may be required by Method SW5035A and should be evaluated according to the specific needs of the client. Consult applicable LIMS program specification.

7.6 To prevent loss of volatile organic compounds, samples must not be opened until the time of analysis.

8. PROCEDURE

Three alternate methods are provided for sample introduction. All internal standards, surrogates, and matrix spikes (when applicable) must be added to samples before purging commences:

- Purge & trap per Method SW5030C (aqueous samples)
- Purge & trap per Method SW5030C (for dilution of solid or waste liquid samples via methanol extraction described in SW5035A)
- Purge & trap per Method SW5035A for solid samples collected in a manner consistent with the method or modification thereof (for samples submitted as samples that must be transferred by laboratory personnel to a purge vessel from containers submitted by the client)

8.1 TYPICAL PURGE & TRAP DEVICE SETTINGS

Instrument conditions may be varied as needed, however, the instrument conditions employed during initial calibration (ICAL) must be used for all subsequent sample analyses that are quantitated using the initial calibration. If operating conditions are altered, a new calibration must be prepared.

Purge & trap settings forArchon/ OI 4560A purge & trap device:
Purge time = 7-11 minutes

Desorb temperature = 190°C
Desorb time = at least 0.5 minute
Trap bake = at least 4 minutes at 210°C. (or according to manufacturer's recommendation for all parameters above)

<u>Purge & trap settings for Teledyne Tekmar Atomx purge & trap device:</u>
Purge time = 7-11 minutes
Desorb temperature = 250°C
Desorb time = at least 0.5 minute
Trap bake = at least 4 minutes at 260°C (or according to manufacturer's recommendation for all parameters above)

8.2 TYPICAL GAS CHROMATOGRAPH SETTINGS

Initial temperature = 50°C
Initial time = 0.1 minute
Temperature ramp A = 10°C/minute
Temperature ramp B = 25°C/minute
Final temperature A = 105°C
Final temperature B = 220°C
Final hold time A = 0 minutes
Final hold time B = until all compounds elute
P&T transfer line temperature = 120°C
GC/MS transfer line temperature = 280°C
Injection temperature = 150°C
Electron energy = 70eV (nominal)
Mass range = 35-270amu
Scan time = 0.6-1 second per scan

8.3 AUTOSAMPLER CLEANING

After use, each purge tube is removed from the autosampler, washed and regenerated per glassware cleaning SOP 334. Additionally, each purge needle is flushed with organic-free DI water (note that the purge tube is rinsed in place, as part of the system program, if using the OI Archon or Atomx autosampler).

8.4 CHROMATOGRAPHIC MAINTENANCE

8.4.1 Bake out the trap and column. Extra blanks may be necessary to achieve an adequate baseline if carryover is observed. Replace trap if performance problems are demonstrated and cannot be alleviated by routine maintenance.

8.4.2 If other chromatographic problems are observed (peak tailing, loss of analytes, poor response, etc.) injection port maintenance (replacement of inlet seal, liner, ferrules, clipping column), MSD source cleaning, etc. may be necessary.

8.4.3 Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Oxidized columns will exhibit baselines that rise rapidly during temperature programming. If a column is oxidized, replacement may be necessary.

8.5 INITIAL CALIBRATION (ICAL)

Instrument conditions may be varied as needed; however, the instrument conditions employed dUring initial calibration must be used for all subsequent sample analyses that are quantitated using that initial calibration. If operating conditions are altered, a new calibration must be prepared.

8.5.1 Each GC/MS system must be hardware-tuned to meet Method criteria (see Table 1 below) for a 5-to-50ng injection or purging of 4-bromofluorobenzene (BFB). A BFB tune is performed prior to analysis to demonstrate the ability of the system to separate ions and assign proper ratios to fragments. Analyses must not begin until these criteria are met. Typically, 1µL of a 50ng/µL BFB tune solution is analyzed by direct injection.

TABLE 1
BFB MASS INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)

MASS	INTENSITY REQUIRED (relative abundance)
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	greater than 50% 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

8.5.2 Set up the purge & trap system as outlined in Method SW5030C, or Method SW5035A if closed system purge & trap analysis is to be utilized. A set of at least five calibration standards containing all of the target analytes and surrogates is needed. The calibration must contain a standard at or below the reporting limit for each compound, the other calibration standards should contain analytes at concentrations that define the range of the method, but do not exceed the linear range of the instrument. Due to the varying reporting limit requirements of the laboratory's clientele and the varying instrument response of the target compounds, eight levels are typically analyzed. Below is a list of typical calibration levels used during ICAL. Project requirements and instrument performance may require modifications to the levels listed (consult applicable LIMS program specifications).

Internal Standard (mg/L of 5 mL purges	**Final Concentration (µg/L of 5mL and 5g purges)**	**Internal Standard (µg/L of 10mL purges)**	**Final Concentration (µg/L of 10mL purges)**
50	160	25	60
50	120	25	40
50	80	25	20
50	60	25	10
50	40	25	4
50	20	25	2
50	10	25	1
50	4	25	0.5
50	2	25	0.25
50	40 CCV level	25	10 CCV level
50	80ICV level	25	20 ICV level

8.5.3 Calibration must be accomplished using the sample introduction technique that will be used for sample analysis. The purging efficiency for 5mL of water is greater than that for 10mL or 25mL. Therefore, develop the standard curve using the volume of sample to be analyzed. Prepare working calibration standards as described in Section 6.

8.5.4 Tabulate the area response of the characteristic ions (see Table 2 at end of SOP) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (A_x\, C_{IS})/(A_{IS}C_x)$$

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_{IS} = Concentration of the specific internal standard.
C_x = Concentration of the compound being measured.

The average RF must be calculated and recorded for each compound using at least five RF values calculated for each compound from the initial calibration curve.

8.5.5 Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. The percent RSD is calculated as follows:

$$\%RSD = \frac{SD}{\overline{RF}_X} x100\%$$

where:

RSD = Relative standard deviation
RF_x = Mean of the initial RFs for a compound
SD = Standard deviation of the initial RFs for a compound

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

where:

RF_i = RF for each of the calibration levels
n = number of RF values (i.e., 7)

8.5.6 LINEARITY

If the %RSD of any compound is <20%, then the compound's response is assumed to be constant over the calibration range, and the average relative response factor may be used for quantification.

If the %RSD of any compound is >20%, a calibration curve of area ratio (A/A_{is}) versus concentration ratio (C/C_{is}), using first or second order regression fit of the five or more calibration points, may be constructed.

The use of calibration curves is a recommended alternative to average response factor calibration and is a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system. The coefficient of determination (COD, r^2 value) of the linear or higher order regression used to define the calibration curve, is an expression of "goodness of fit", and must be $\geq$0.99.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weigh the curve to either the inverse of the concentration (or, more accurately, the concentration *ratio*) or to the inverse of the square of the concentration.

Quadratic regressions may be used with a minimum of 6 calibration points following the guidelines in SW-846 Method 8000C, and must yield a COD (r^2 value) of $\geq$0.99. A quadratic regression should not be used to compensate for detector saturation.

The type of curve fit applied should be chosen to best represent the data.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

8.5.7 Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. Client calibration requirements may also be prescribed in the LIMS program specification.

If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99 for r^2 value) for the alternative curve fits, then the chromatographic system is considered too imprecise for analysis (11.3.4.2 – 8260C).

8.5.8 It is recommended that a minimum response factor for the most common target analytes as noted in Table 3, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. ALS demonstrates this sensitivity at the reporting limits in each batch with a reporting limit verification sample (RVS). See section 9.7.

8.6 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is analyzed after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time.

8.6.1 The percent difference for each analyte considered to have an adequate response by preparation technique 5030/5035 (i.e. not a poor purger, high temperature requirement, etc.) for method 8260 (revision 3, August 2006) must be within 30%, allowing for up to two analytes to exceed the 30% criteria. Target analytes which exceed the 30% criteria are considered estimates. Documentation in the associated case narrative, and inclusion of the response factor calibration report (EPA Form 7) shall be considered sufficient client notification.

The second source check can also serve as the laboratory control sample (LCS) for samples analyzed in the same 12 hour shift as the ICAL. The LCS criteria may be different than the ICV criteria described above.

8.7 CONTINUING CALIBRATION VERIFICATION (CCV)

The ICAL curve for each compound of interest must be checked and verified once every 12 hours during analysis with the introduction technique used for samples. This is accomplished by analyzing a calibration standard (CCV) that is at or near the midpoint concentration for the working range of the GC/MS at the beginning of each 12-hour sequence when initial calibration is not performed.

8.7.1 Prior to the analysis of samples, inject or purge 50mg of the 4-bromofluorobenzene standard following Method SW5030C or Method SW5035A. The resultant mass spectra for the BFB must meet all of the criteria given in Table 1 (shown previously) before sample analysis begins. These criteria must be met at the start of each 12-hour shift.

For the CCV analysis, the %D for all target compounds are evaluated against the initial calibration.

If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds may fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples (11.4.5.4 – 8260C). In cases where compounds fail, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values, or the associated samples re-analyzed. No compounds may exceed the 20% D criteria for EPA Method 624

Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

- When the acceptance criteria for the continuing calibration verification are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted, or:

- When the acceptance criteria for the continuing calibration verification are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level, if acceptable to client/project. Otherwise the samples affected by the unacceptable verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

8.7.2 RETENTION TIME REPRODUCIBILITY

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard level of the most recent initial calibration, the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from that in the midpoint standard level of the most recent initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. Samples should not be analyzed and reported if the criteria described above are not met.

8.8 SAMPLE ANALYSIS

BFB tuning criteria and calibration verification criteria (discussed above) must be met before analyzing samples. All samples and working standard solutions must be allowed to warm to ambient temperature before analysis. Set up the purge & trap system as outlined in Method SW5030C, or Method SW5035A if closed system purge & trap introduction will be used.

8.8.1 PURGE TEMPERATURE

8.8.1.1 For soil analysis, the ICAL, all CCVs, and all field and QC samples shall be heated to 40°C during the purge.

8.8.1.2 For aqueous analysis, a heated purge is not required. The same purge conditions used for soil analysis may be used for aqueous analysis, however, if the ICAL, all CCVs, and all field and QC samples are heated to 40°C during the purge.

It is recommended that purge volumes of 10 to 25mL should not use a heated purge due to the amount of water vapor that may be introduced into the purge & trap system. The ICAL, all CCVs, and all field and QC samples should be left at ambient temperature during the purge.

8.8.2 AQUEOUS ANALYSIS

8.8.2.1 Allow all aqueous samples to come to ambient temperature prior to analysis. All working standards and some sample dilutions are prepared in 50mL volumetric flasks, spiked accordingly, then transferred to a 40mL VOA vial (without headspace). The 40mL sample vials are then placed in the autosampler carrousel. The autosampler is programmed to remove the appropriate sample volume (usually 10mL), add internal standards and surrogates, and proceed with the purge and trap procedure.

8.8.2.2 The process of taking an aliquot destroys the validity of aqueous samples for future analysis; therefore, if there is only one VOA vial, the analyst should prepare a second aliquot for analysis concurrently to protect against possible loss of sample integrity, or transfer the remaining sample to a 20mL VOA vial (without headspace) and refrigerate. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly.

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8.8.2.3 When a sample is analyzed that has saturated ions from a high concentration compound, this analysis must be followed by an organic-free reagent water blank analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences (refer to Section 4 for further details).

8.8.2.4 The following procedure is appropriate for diluting aqueous purgeable samples. Sample dilution is based on analyte concentration, non-target compound concentration, or the presence of surfactants (foaming samples). All steps must be performed without delay until the diluted sample is in a gas-tight syringe. If usable data has not been generated for a less diluted analysis, the dilution should keep the response of the major constituents (previously saturated peaks) in the upper portion of the linear range to generate the lowest reporting limits possible.

Dilutions may be made in volumetric flasks (10 to 100mL) or gas-tight syringes (5mL or 30mL). Select the volumetric flask or syringe that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions

Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

Inject the proper aliquot of sample from the syringe into the flask. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert three times. The sample is now ready for analysis.

8.8.2.5 The following procedure can be used to composite aqueous samples prior to GC/MS analysis:

The sample must be at 0 to 6°C during this step to minimize volatilization losses. Combine equal portions of the samples to a chilled volumetric flask. Invert the flask 3 times and transfer to an appropriate container for storage or analysis

8.8.3 SOIL SAMPLE ANALYSIS BY METHOD SW5035A

8.8.3.1 Homogenize the sample well, taking care to minimize the loss of volatile constituents.

8.8.3.2 Weigh 5g of soil into an appropriate purge vessel; place the sample on the autosampler. For method blanks and LCSs, 5g of Ottawa sand should be added to the purge vessel.

8.8.3.3 Add 5mL of organic-free water to the sample. In the case of LCS or MS samples, the associated spike is added with this aliquot.

8.8.3.4 The Archon autosampler (or equivalent) adds a total of 5mL of reagent containing internal standards and surrogates to each sample. The sample is now taken through the purge and trap procedure.

8.8.3.5 The following procedure is appropriate for diluting soil purgeable samples. Soil sample dilution is based on analyte concentration or unknown compound concentration. If usable data has not been generated for a less diluted analysis, the dilution should keep the response of the major constituents (previously saturated peaks) in the upper portion of the linear range to generate the lowest reporting limits.

Soil dilutions are made by weighing an aliquot of less than 5g of sample into the purge tube. To ensure a representative sample aliquot, no less than 0.5g of soil should be purged. For reporting purposes, a nominal amount of 5g will be considered the purge amount, and amounts less than this will be treated as dilutions.

8.8.4 MEDIUM LEVEL SOIL SAMPLES (METHANOL-EXTRACTION) Methanolic extraction /analysis is used for high concentration solid samples requiring dilutions greater than that which can be soundly achieved using smaller sample volume, or for samples that are difficult to homogenize.

8.8.4.1 Homogenize the sample as well as possible, taking care to minimize the loss of volatile constituents.

8.8.4.2 Weigh approximately 5g (record actual weight to 0.01g) of sample into a labeled, tared 20mL VOA vial. Clean the outer lip of the vial with a Kimwipes^{TM} before obtaining the final weight. In some instances, such as low density soils or odd matrices, an aliquot of less than 5g may be necessary.

8.8.4.3 Add 5mL of methanol, cap and shake vigorously for 2 minutes. Allow solid and methanol to separate for at least 10 minutes. Note that alternate soil weights and methanol volumes may be used depending upon the level of sample dilution required. Enough methanol must be added to the vial to completely cover the soil aliquot.

8.8.4.4 Calculate the volume of the methanol extract that when brought to a final volume of 5mL in water, will bring the dilution concentration into the upper portion of the instrument calibration (factor in any dilution that may have been made by the initial extraction of the sample with methanol). To protect the system from trap or column overload, a maximum of 100µL of extract may be used. Proceed with the analysis as discussed for aqueous samples above (Section 8.8.2).

8.8.4.5 A medium level blank should be prepared in the same manner using 5.0g of Ottawa sand and 5mL of methanol. 100µL of this methanol extract injected into 5mL of water is to be analyzed before the sample extract, to ensure no methanol contamination.

8.8.5 SOIL SAMPLE ANALYSIS BY METHOD 5035A

8.8.5.1 Transfer the contents of an EnCore™ (or equivalent) soil sampler to a 40mL VOA vial containing a magnetic stir bar.

8.8.5.2 Use 5g of Ottawa sand in a 40mL VOA vial as the matrix basis for method blanks (MBs)and laboratory control samples (LCSs).

8.8.5.3 Add 5mL of organic-free water to the vial.

8.8.5.4 For matrix spikes, add 2µL (or appropriate amount) of intermediate spiking solution.

8.8.5.5 Samples may be submitted by clients in 40mL vials which already contain water, preservative ($NaHSO_4$) and stir bar or water and stir bar only. Samples submitted in vials are analyzed in the vials without opening the vial.

8.8.5.6 Place vial on the autosampler.

8.8.5.7 The Archon (or equivalent) is used to add internal standards and surrogates solution and 5mL of organic-free water to the purge vessel bringing the final liquid purge volume to 10mL.

8.8.5.8 Place the VOA vial in the Archon autosampler which will automatically inject 1µL of surrogates and internal standards (if appropriate) prior to purging. <u>Note</u>: The 1µL volume is approximated; as instructed by the instrument manufacturer, the internal loop used to deliver the standard is calibrated for each autosampler to determine the absolute volume being delivered. The autosampler will stir and heat the contents of the VOA vial during the purge process.

8.8.5.9 Soil dilutions are made by weighing an aliquot of less than 5g from the Encore™ (or equivalent) into the VOA vial, if acceptable per client or project. To ensure a representative sample aliquot, no less than 0.5g of soil should be purged. For reporting purposes, a nominal amount of 5g will be considered the purge amount, and amounts less than this will be treated as dilutions. If a dilution greater than can be obtained by 0.5g of soil is required, a medium level extraction must be performed by extracting the contents of the EnCore™ as described in Section 8.8.4 above.

8.9 DATA INTERPRETATION

8.9.1 QUALITATIVE ANALYSIS

8.9.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of the method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met:

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

The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component. (RRT = RT of the analyte/ RT of the internal standard).

The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than

25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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

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

8.9.1.2 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 tentatively identified compound (TIC) determination will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20%. Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.

Molecular ions present in the reference spectrum should be present in the sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the analyst assign a tentative identification.

8.9.2 QUANTITATIVE ANALYSIS

8.9.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. The IS used shall be the one nearest the retention time of that of a given analyte.

8.9.2.2 When the detector response is linear and passes through the origin, calculate the concentration of each identified analyte in the sample as follows:

<u>WATER</u>:

$$Concentration(\mu g/L) = \frac{(A_x)(I_s)}{(A_{IS})(\overline{RF})(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured
I_s = Amount of internal standard injected (ng)
A_{IS} = Area of characteristic ion for the internal standard
$\overline{RF}$ = Mean relative response factor for compound being measured
V_o = Volume of water purged (mL), taking into consideration any dilutions made

<u>SEDIMENT/SOIL SLUDGE</u> (on a dry-weight basis) & <u>WASTE</u> (normally on a wet-weight basis):

$$Concentration(\mu g/kg) = \frac{(A_x)(I_s)V_t)}{(A_{is})(\overline{RF})(V_i)(W_s)(D)}$$

where:

A_x, I_s, A_{is}, $\overline{RF}$ = Same as for water.
V_t = Volume of total extract (µL) (Use 10,000µL or a factor of this when dilutions are made)

Vi = Volume of extract added (µL) for purging
WS = Weight of sample extracted or purged (g)
D = % dry weight of sample/100, or 1 for a wet-weight basis

8.9.2.3 Where requested by the client, an estimate of concentration for non-calibrated components in the sample may be made. The formulae given above should be used with the following modifications: The areas A_x and A_{IS} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. The chromatographic data system calculates the concentration and reports which IS was used in the calculation. Use the nearest IS free of interferences. Upon request, ALS will report the top 10 non-calibrated components (Tentatively Identified Compounds, TICs) with total ion areas > 10% of the total ion area of the nearest internal standard. Identification of TICs with less than 10% relative abundance is difficult at best, and generally should not be attempted. Some clients may request the reporting of more compounds or compounds with lower areas relative to the closest IS. Consult LIMS program specification for further direction.

8.9.2.4 Alternatively, the regression line fitted to the initial calibration may be used for determination of analyte concentration.

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

For this method, an analysis batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specification for additional or alternative requirements.

9.2 BLANK ANALYSIS

A method (reagent) blank (MB) must be analyzed for each 12-hour BFB tune and per batch of 20 or fewer field samples of similar matrix. Target compounds may not be detected above one-half the reporting limit (RL); or as otherwise stipulated in the applicable LIMS program specification. Common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride) are allowed at levels as high as the RL; or as otherwise stipulated in the applicable LIMS program specification.

Occurrence of these common laboratory contaminants should be considered a warning and must be reported in the data package case narrative. See QC Table for further details.

9.3 SURROGATES
Surrogate recovery is monitored to assess method performance of the particular matrix. Surrogates are added to all standards, blanks, samples and QC samples prior to analysis. See QC Table for acceptance limits and corrective actions.

9.4 INTERNAL STANDARDS
Internal standards are added to all standards, field and quality control samples analyzed. Retention times and responses are evaluated for internal standards. See QC Table for acceptance limits and corrective actions.

9.5 LABORATORY CONTROL SAMPLES
A matrix-specific laboratory control sample (LCS) is analyzed per batch of 20 field samples. It is ALS's practice to also analyze a laboratory control sample duplicate (LCSD) per batch of 20 field samples. LCS (LCSD) samples are analyzed to evaluate the efficiency of the method performed. See QC Table for acceptance limits and corrective actions.

9.6 MATRIX SPIKE(S)
A matrix spike (MS) and matrix spike duplicate (MSD) sample are analyzed to evaluate the effect of the matrix. Additional sample volume of client samples is needed to perform these analyses. The frequency of the MS/MSD shall be one pair per batch of 20 field samples, assuming adequate volume has been provided. See QC Table for acceptance limits and corrective actions.

9.7 DETECTION LIMITS MDL/DL limits determinations are completed annually and as defined by the reference method. A MDL/DL study must also be performed as a component of method validation or whenever the basic chemistry of a procedure changes. See ALS SOP 329 for guidance on detection limits. ALS uses RVS samples run with each batch to assess the method sensitivity on an ongoing basis and to calculate detection limits as needed.

10. DEVIATIONS FROM METHOD

This SOP meets the requirements of Method SW8260C. Alternate quantitation ions may be used to limit or eliminate common interferences caused by co-elution of standards or matrix contributions.

EPA METHOD 624 REQUIREMENTS

10.1 Suggested surrogates and internal standards are listed in EPA 624, Table 3. ALS uses the same surrogates and internal standards for both Methods SW8260C and EPA 624 as follows: ISs - fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4; SSs - toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4 and dibromofluoromethane. Two of each of the SSs and ISs listed above are included in EPA 624, Table 3.

10.2 Method EPA 624 states that the concentration of the surrogate spike used should be 30µg/L; ALS typically uses a 50µg/L concentration surrogate spike.

10.3 EPA 624 states specific adsorbent trap and purge & trap conditions, and chromatographic columns and conditions, as well as mass spectrometer conditions to be used in the execution of the method (i.e., specific purge time, use of a packed column, and scanning conditions tailored for packed column use). Some of these materials, apparatus, and conditions have been eclipsed by technology as described in this SOP. Note that Section 8.1.2 of Method EPA 624 provides for the use of technological advances so long as the precision and accuracy requirements put forth by the Method can be achieved.

10.4 Method EPA 624 requires at least three points in the ICAL; ALS quantitates from a 5-to-7-point curve to meet compliance requirements for Methods SW8260C. This approach also meets compliance requirements for EPA 624, as more than three points are used to calibrate.

10.5 Method EPA 624 states that if the %RSD of the average response factor is less than 35%, then an average response factor may be used. Otherwise, construct a linear curve with a correlation coefficient greater than 0.995. ALS follows the calibration criteria discussed previously in the SOP.

10.6 EPA 624 specifies that the BFB tune period is 24 hours. ALS follows the procedure as discussed in SW8260 C, which specifies that BFB criteria must be passed every 12 hours.

10.7 Method 624 states that a continuing calibration verification (CCV) must be performed every working day (i.e., every 24 hours). ALS observes a criterion that a CCV must be performed every 12 hours. Method 624 also requires that the results of the CCV must meet the requirements set forth in Table 5 of the Method, and that any compounds without limits in this Table must have their recovery reported, but corrective actions are not required.

10.8 EPA 624 states that a matrix spike (MS) and laboratory control spike (LCS) must be performed per every 20 samples. The native sample only needs to be spiked once; a matrix spike duplicate (MSD) sample is not required. EPA 624 also states that the matrix spikes and laboratory control (blank) spikes must meet the acceptance criteria listed in Table 5 of the Method (note that not all compounds have acceptance limits in this Table; for these compounds, the recovery must be reported, however, corrective actions based on those results are not required). Furthermore, EPA 624 discusses matching each compound's spike amount with the amount of the compound in the samples chosen for spiking, and also matching the spike amount to the appropriate regulatory level for each compound. Because samples from several sites are usually batched together, it is ALS's practice to use only one spiking level is for each compound.

10.9 Method EPA 624 states that a set of 4 QC Check samples must be analyzed by an analyst before any samples are processed to demonstrate the ability to perform the method. The concentrations of each compound must be 20µg/L, and the results must fall within the acceptance criteria specified in Table 5 of the method. ALS does observe Demonstration of Capability (DOC) requirements, but at the spike levels presented in the SOP and requiring that the results must meet the laboratory's LCS criteria established for the procedure (based on SW-846 guidance).

10.10 All 8260C initial and continuing calibration criteria meets or exceeds EPA 624 criteria. There are no allowances for compounds exceeding calibration criteria for EPA 624

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water, Method 524.2, USEPA, Office of Research Development, Environmental Monitoring and Support Laboratory, Cincinnati, OH, 1986.

12.2 40 CFR, Part 136, Appendix A, 7-1-86 Edition, Method 624.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, “Method 8260C”, Revision 3, August 2006.

12.4 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 5030C, “Purge And Trap For Aqueous Samples”, Revision 3, May 2003.

12.5 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 5035A, “Closed System Purge And Trap And Extraction For Volatile Organics in Soil And Waste Samples”, Revision 1, July 2002.

12.6 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 8000C, “Determinative Chromatographic Separations”, Revision 3, March 2003.

TABLE 2 CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS		
TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
dichlorodifluoromethane	85	87
chloromethane	50	52
vinyl chloride	62	64
bromomethane	96	94
chloroethane	64	66
trichlorofluoromethane	101	151, 153
acrolein	56	55, 58
1,1-dichloroethene	96	53, 61
1,1,2-trichloro-1,2,2-trifluoroethane	101	103, 151, 153
acetone	58	43
iodomethane	142	127, 141
carbon disulfide	76	78
methylene chloride	84	86, 49
trans-1,2-dichloroethene	96	61, 98
methyl tertiary butyl ether	73	57
acrylonitrile	53	52, 51
1,1-dichloroethane	63	65, 83
vinyl acetate	43	86
cis-1,2-dichloroethene	96	61, 98
2-butanone	43	72
bromochloromethane	128	49, 130
chloroform	83	85
1,1,1-trichloroethane	97	99, 61
2,2-dichloropropane	77	97
carbon tetrachloride	117	119
1,1-dichloropropene	75	110, 77
1,2-dichloroethane	62	98
benzene	78	52, 77
trichloroethene	95	97, 130, 132
1,2-dichloropropane	63	112
dibromomethane	93	95, 174
bromodichloromethane	83	85, 127
2-chloroethyl vinyl ether	63	65, 106
cis-1,3-dichloropropene	75	77, 39
4-methyl-2-pentanone	43	58, 85, 100

TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
toluene	91	92
trans-1,3-dichloropropene	75	77, 39
1,1,2-trichloroethane	83	85, 97
2-hexanone	43	58, 57, 100
tetrachloroethene	164	129, 131, 166
1,3-dichloropropane	76	78
dibromochloromethane	129	127
1,2-dibromoethane	107	109, 188
1-chlorohexane	91	55, 93
chlorobenzene	112	77, 114
1,1,1,2-tetrachloroethane	131	133, 119
ethylbenzene	91	106
m- + p-xylene	106	91
o-xylene	106	91
styrene	104	78
bromoform	173	175, 254
isopropylbenzene	105	120
1,2,3-trichloropropane	110	75, 77
1,1,2,2-tetrachloroethane	83	131, 85
bromobenzene	156	77, 158
n-propylbenzene	91	120
2-chlorotoluene	91	126
1,3,5-trimethylbenzene	105	120
4-chlorotoluene	91	126
tert-butylbenzene	119	91, 134
1,2,4-trimethylbenzene	105	120
sec-butylbenzene	105	134
1,3-dichlorobenzene	146	111, 148
p-isopropyltoluene	119	134, 91
1,4-dichlorobenzene	146	111, 148
n-butylbenzene	91	92, 134
1,2-dichlorobenzene	146	111, 148
1,2-dibromo-3-chloropropane	75	155, 157
1,2,4-trichlorobenzene	180	182, 145
hexachlorobutadiene	225	223, 227
naphthalene	128	

TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
1,2,3-trichlorobenzene	180	182, 145
trans-1,4-Dichloro-2-butene	53	88, 75
1,1,1,2-tetrachlorobenzene	131	133, 119
1,4-dioxane	88	58, 43, 57
acetonitrile	41	40, 39
allyl chloride	76	41, 39, 78
chloroprene	53	88, 90, 50
cis-1,4-dichloro-2-butene	75	53, 77, 124
ethanol	45	46, 43
ethyl methacrylate	69	41, 99, 86
ethyl-tert-butyl ether	59	87, 57, 41
hexachloroethane	201	166, 199, 203
isobutyl alcohol	43	41, 42, 74
isopropyl ether	45	43, 87, 59
methacrylonitrile	41	67, 39, 52
methyl methacrylate	69	41, 100, 39
pentachloroethane	167	130, 132, 165
propionitrile	54	52, 55, 40
tert-amyl methyl ether	73	87, 55, 71
tert-butanol	59	41, 57, 43
1,2-dichloroethane-d_4 (SUR)	65	
toluene-d8 (SUR)	98	
4-bromofluorobenzene (SUR)	95	174, 176
dibromofluorobenzene (SUR)	113	
chlorobenzene-d_5 (IS)	82	117
1,4-dichlorobenzene-d_4 (IS)	152	115,150
1,4-difluorobenzene (IS)	114	
fluorobenzene (IS)	96	70

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	0.100	0.254
Trichloroflouromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-triflouroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfate	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethane	0.200	0.655
cis-1,2-Dichloroethane	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	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	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.652
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.916

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
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-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.

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds	**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**

QC Check	Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 12 hour period	BFB abundance criteria (Table 1) must be met	Re-tune. Do not proceed with analysis until tune meets criteria.
Initial Calibration (ICAL)	Prior to sample analysis.	Ave RF may be used if: analytes are ≤20% RSD r^2 for regression (or quadratic) curve fit must be ≥0.99; a quadratic curve may be used if 6 or more data points are used	When client or method criteria are not met, reanalyze ICAL. Evaluate/correct instrument malfunction if required
Initial Calibration Verification (ICV): different source than that of ICAL standards	Following every ICAL	Measured concentrations of all analytes should be within 30% of expected concentrations. Sporadic failures allowed for up to two analytes IS retention times <30 seconds drift from mid-point in most recent ICAL IS areas –50 to +100% of corresponding internal standard area in the mid-point of the most recent ICAL	Re-analyze ICV. If still out, evaluate/correct instrument malfunction as needed; perform a new ICAL
Continuing Calibration Verification (CCV); at or near mid-point	Every 12-hour period following tune, if ICAL not performed. Required for quantitating all samples analyzed during the 12 hour sequence	Analytes should be within 20% of expected concentrations. • See section 8.7.2 IS retention times <30 seconds drift from mid-point in most recent ICAL IS areas –50 to +100% of corresponding internal standard areas in the mid-point of the most recent ICAL	Re-analyze the daily standard. If failure repeats, evaluate/correct instrument malfunction; perform a new ICAL NOTE: Recoveries that are high and outside of the stated acceptance criteria may be acceptable in some programs if the analyte that is high was not detected in the associated samples.
Method Blank (MB)	Every 12-hour period; after each calibration/check and 1 per batch of 20 samples of like matrix	< ½ RL for all target compounds, except common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride), which are allowable to the RL; or as otherwise stipulated in the applicable LIMS program specification.	Re-analyze to determine if instrument contamination was the cause. If MB is still non-compliant, correct the problem and obtain a successful MB analysis before resuming analysis of samples. NOTE: Reporting of samples associated with MBs that yield contaminants may be permitted by some program specifications or at the client's discretion. Example: Toluene in MB at RL but not detected in any sample above the MDL. In this case, document occurrence and resolution using a Nonconformance Report (NCR), SOP 928.

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds	**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**

QC Check	Frequency	Acceptance Criteria	Corrective Action
Surrogates (SS)	Every standard, client sample and QC sample	See laboratory or other applicable limits; recoveries should be within these limits	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, sample may be reanalyzed once (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If still non-compliant, report results and narrate. If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon content contributes to SS areas), reanalysis is not required. Narrate NOTE: Per program specifications, surrogate recovery that is high and outside of acceptance criteria, with no associated target compounds detected, may not require reanalysis.
Internal Standard (IS)	Every standard, client sample and QC sample	Average area within -50% to +100% window of corresponding daily calibration verification standard area RT shift <30 seconds compared to daily standard ; relative retention time (RRT) of sample must be ± 0.06 RRT units of standard	Inspect instrument for malfunction, correct. Sample may be reanalyzed (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon content contributes to IS areas), reanalysis is not required. Narrate.
Matrix Spike (MS)	1 per batch of 20 samples of like matrix	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause. Narrate.
Matrix Spike Duplicate (MSD) or Duplicate	1 per batch of 20 samples of like matrix	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits RPDs for the spiked compounds should also be within advisory limits	If non-compliant, check calculations for errors. If significant differences exist between the duplicate results, consult with Department Manager (reanalysis of the sample and spikes may be necessary, or sample inhomogeniety may be the likely cause).

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS) or Duplicate	1 per batch of 20 samples of like matrix; typically the LCSD is analyzed when matrix spikes are not performed	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these limits NOTE: When the full list of compounds is spiked, the laboratory will accept a small number of sporadic marginal exceedances, based on the probability that a certain number of compounds will exceed their control limits. Exceedances must be sporadic and marginal, systematic or gross failures shall not be accepted.	If non-compliant, check calculations and spike preparation for documentable errors; correct as needed. If no errors are found, then re-analyze to determine if instrumental conditions was the cause. Notify the Supervisor and initiate corrective action (NCR)if needed. Re-analyze associated samples, if appropriate. Note that recoveries that are high and outside of acceptance criteria may be acceptable, when the same target compound is not detected in any sample in the batch. Narrate.
RVS	Per Batch	Value should be greater than ½ RL	Not used for batch evaluation unless specified by client requirements.

ALS Standard Operating Procedure

DOCUMENT TITLE:	1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN AQUEOUS SAMPLES BY MICROEXTRACTION AND GAS CHROMATOGRAPHY
REFERENCED METHOD:	EPA 8011
SOP ID:	SOC-8011
REVISION NUMBER:	1
EFFECTIVE DATE:	2/26/16

ALS Environmental

ALS-Kelso SOP Annual Review Statement

SOP Code: SOC-8011
Revision: 1

An annual review of the SOP listed was completed on (date): 7/8/17

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: LEP   Date: 7/8/17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN AQUEOUS SAMPLES BY MICROEXTRACTION AND GAS CHROMATOGRAPHY

ALS-KELSO

SOP ID:	SOC-8011	Rev. Number:	1	Effective Date:	2/26/2016

Approved By: Date: 2/4/16
Department Supervisor/Technical Director – Loren Portwood

Approved By: Date: 2/5/16
QA Manager – Carl Degner

Approved By: Date: 2/8/16
Laboratory Director – Jeff Grindstaff

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ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

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1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN AQUEOUS SAMPLES BY MICROEXTRACTION AND GAS CHROMATOGRAPHY

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the procedure used for the analysis of 1,2-Dibromoethane (EDB) and 1,2-Dibromo-Chloropropane (DBCP) by micro-extraction and gas chromatography using Method 8011. This procedure describes both the preparation and analysis procedures used to determine the target analytes and reporting limits listed.

1.2. This procedure is used to determine the analytes of interest in aqueous (water) samples, excluding drinking water. The Method Reporting Limits (MRLs) and Method Detection Limits (MDLs) for target analytes are presented in Table 1.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD) may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. A 35mL sample aliquot is extracted with 2mL of hexane. An aliquot (5 uL) of extract is injected onto a GC equipped with an electron capture detector (ECD). Identification is based on retention time. Quantitation is performed using an external standard calibration technique using aqueous standards prepared in the identical manner as the sample.

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc…) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. Independent Calibration Verification (ICV) - Initial calibration verification standards, which are analyzed after initial calibration but prior to sample analysis, in order to verify the validity of the standards used in calibration. The ICV standards are prepared from materials obtained from a source different from that used to prepare calibration standards.

3.3. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision.

3.4. Standard Curve - A standard curve is a calibration curve, which plots concentrations of a known analyte standard versus the instrument response to the analyte. The appropriate criteria for assessing the validity of the calibration curve must be followed prior to quantitation of target analytes in actual sample analyses.

3.5. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.7. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.8. Laboratory Control Sample (LCS) - The laboratory control sample is an artificial sample composed of analyte free solid matrix which is spiked with a known concentration of analytes of interest. For this method, the laboratory fortified blank (LFB) may fulfill the LCS requirement.

4. INTERFERENCES

4.1. Impurities contained in the extracting solvent may account for problems with interferences. Solvent blanks should be analyzed on each new lot of solvent before use. Monitoring the method blanks also checks the extracting solvent. Whenever interferences are noted in the method blank, the analyst should retest the extracting solvent. It may be necessary to obtain a new source of solvent. Alternatively, low-level interferences generally can be removed by distillation or column chromatography. Protect interference-free solvents by storing in an area free of organochlorine solvents.

4.2. This liquid/liquid extraction technique efficiently extracts a wide boiling range of non-polar organic compounds and, in addition, extracts polar organic components of the sample with varying efficiencies. These co-extracted materials may interfere with the chromatographic determination. Low concentrations of EDB may be masked by very high levels of dibromochloromethane (DBCM), a common disinfection byproduct of chlorinated drinking waters. A DBCM standard should be analyzed periodically to establish resolution between EDB and DBCM.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. EDB and DBCP have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Sample Collection

6.1.1. Collect all samples in 40mL VOA vials into which 3mg of sodium thiosulfate crystals have been added just prior to shipping to the sampling site. Alternately, 75 µL of freshly prepared sodium thiosulfate solution (40 mg/mL) may be added to empty 40mL bottles just prior to sample collection.

6.1.2. Field blanks should be handled along with each sample set, which is composed of the samples collected from the same general sampling site at approximately the same time. At the laboratory, fill a minimum of two sample bottles with reagent water, seal, and ship to the sampling site along with sample bottles. Wherever a set of samples is shipped and stored, it must be accompanied by field blanks.

6.2. Sample Preservation and Storage

6.2.1. A dechlorinating agent (sodium thiosulfate) must be added to each sample to avoid the possibility of reactions that may occur between residual chlorine and indeterminate contaminants present in some solvents, yielding compounds that may subsequently interfere with the analysis. The presence of sodium thiosulfate will arrest the formation of DBCM.

6.2.2. Samples must be iced or refrigerated at 4 ± 2ºC from time of collection until extraction. The sample storage area must be free of organic solvent vapors.

6.2.3. Samples must be extracted within 14 days of collection. Samples not extracted within this period must be discarded and replaced. Because of the potential for solvent evaporation, it is preferred that extracts be analyzed immediately following preparation. When necessary, extracts may be stored in tightly capped vials at 4 ± 2ºC or less for up to 24 hours.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagents

7.1.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to ADM-RTL, *Reagent/Standards Login and Tracking* for the complete procedure and documentation requirements.

7.1.2. Sodium Chloride, NaCl, ACS reagent grade. This should be pulverized and heated in a muffle furnace at 400° for 30 minutes prior to use.

7.1.3. Sodium thiosulfate, $Na_2S_2O_3$ -- ACS reagent grade, for preparation of solution (40 mg/mL), dissolve 1 g of $Na_2S_2O_3$ in reagent water and bring to 25 mL volume in a volumetric flask.

7.1.4. Methanol, pesticide grade.

7.1.5. Hexane, pesticide grade.

7.2. Standards

7.2.1. Stock standard solutions may be purchased from a number of vendors. All standards purchased from vendors must be traceable to NIST or A2LA certified reference materials. The vendor-assigned expiration date is used. Stock solutions are purchased from Ultra Scientific, Accustandard, or equivalent.

7.2.2. A working (intermediate) standard is prepared from the stock standards and containing each analyte in methanol. The intermediate standard should be prepared at a concentration that can be easily used to prepare the calibration standards.

7.2.3. The calibration standards are extracted using the procedure in section 11.0. The analyst prepares a minimum 5-point calibration curve containing each target analyte using the working standard. The nominal concentrations of the standards are 0.075, 0.125, 0.25, 0.625, 1.25, 3.75, 5.0, and 10 ug/L. The CCV is prepared with each extraction batch to demonstrate that the initial calibration is acceptable.

7.2.4. The ICV standards are prepared from materials obtained from a source different from that used to prepare calibration standards, and extracted using the procedure in section 11.0. The ICV is extracted in the same batch as the calibration standards and analyzed following the calibration and before any sample analysis.

7.2.5. A matrix spike solution is prepared from the stock solution in methanol. This solution is stored in the refrigerator for up to one month. Solutions may be stored for up to one month as long as the stability of the solution is demonstrated.

7.2.6. Store all standards in a manner that prevents loss of analytes or degradation (e.g. with minimal headspace) and following ALS guidelines for expiration periods. Calibration standards should be prepared fresh, prior to use.

8. APPARATUS AND EQUIPMENT

8.1. Sample Containers -- 40mL screw cap VOA vials with Teflon™-lined caps. Individual vials shown to contain at least 40.0mL can be calibrated at the 35.0mL mark so that volumetric, rather than gravimetric, measurements of sample volumes can be performed. Pre-cleaned vials may be purchased. Alternatively, wash vials and septa with detergent and rinse with tap and distilled water. Allow the vials and septa to air dry at room temperature, place in a 105°C oven for one hour, then remove and allow to cool in an area free of organic solvent vapors.

8.2. GC Instrumentation

8.2.1. Gas Chromatograph, equipped with cool-on-column or split/splitless injection port that is temperature programmable with an ECD, Agilent 6890 or 7890.

8.2.2. Autosampler, capable of reproducible 5.0 µL injections, Agilent 7683

8.2.3. Columns, J&W Scientific or equivalent columns are used;
Column 1: Rtx-CLPesticides 30m x 0.32mm ID, 0.50 um df
Column 2: Rtx-CLPesticides II 30m x 0.32mm ID, 0.25 um df

8.2.4. Data system, compatible with detectors and capable of measuring peak areas and retention times, Agilent Enviroquant.

8.3. Vials -- auto sampler, crimp top or screw cap with Teflon™ faced septa, 1.8mL.

8.4. Micro Syringes – Various sizes.

8.5. Disposable Pipettes -- 2.0mL and 5.0mL transfer.

8.6. Standard Solution Storage Containers -- bottles with Teflon™ lined screw caps.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Inline purifiers or scrubbers should be in place for all sources of carrier gas or detector gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, O-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the guard column or analytical column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool.

9.3.3. The autosampler should be cleaned periodically. This includes turret cleaning and cleaning or replacing the syringe. Refer to manufacturer’s instructions for autosampler restarting.

9.3.4. The detector should be leak-checked and serviced as specified by the manufacturer.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in ADM-TRAIN, *ALS-Kelso Training Procedure*. Documenting method proficiency, as described in this SOP, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. For samples and field blanks contained in 40 mL VOA vials, remove the container cap. Discard a 5mL volume using a 5mL transfer pipette or 10 mL graduated cylinder. Weigh the container with contents to the nearest 0.1 g and record this weight on the benchsheet for subsequent sample volume determination. Deionized water (35mL) is used for method blanks, lab control samples and standards.

11.1.2. Add matrix spike to appropriate vessels as listed in 11.1.3. Add approximately 7 grams of muffled NaCl to the samples. Add 2ml of hexane to each extraction vessel. After replacing the cap, the sample is shaken vigorously for 2 minutes. The sample is allowed to settle for approximately 5 minutes. The hexane layer is placed in a 2 ml autosampler vial for GC analysis. The water is emptied and the sample vial is weighed to determine the sample volume extracted.

11.1.3. Aqueous standards (if needed), LCS, MS and CCVs are prepared such that the final concentrations of the final extract are as follows:

	Final Concentration	Amt. of 50 ug/L spike Solution added.
Cal level 1	0.075 ug/L	3 ul
Cal level 2	0.125 ug/L	5 ul
Cal level 3	0.250 ug/L	10 ul
Cal level 4	0.625 ug/L	25 ul
Cal level 5	1.25 ug/L	50 ul
Cal level 6	3.75 ug/L	150 ul
Cal level 7	5.00 ug/L	200 ul
Cal level 8	10.0 ug/L	400 ul
ICV	1.25 ug/L	50 ul
LCS	4.375 ug/L	175 ul
MS	4.375 ug/L	175 ul

CCV	1.25 ug/L	50 ul

11.1.4. All calibration standards, LCS, MS, CCVs and MDL checks are prepared by extracting in the same manner as samples.

11.2. Analysis

11.2.1. Establish the operating parameters on the instrument as follows:

Inlet: Splitless
Inlet temperature: 100ºC for 0.25 min., 250ºC/min. to 250ºC for 10min
Injection volume - 5 uL
Flow rate - constant flow mode at 2.3 mL/min.
Temperature program - Initial 40ºC and hold 3.0 min.
- program at 3ºC/min. to 60ºC with no hold
- program at 25ºC /min. to 85ºC with no hold
- program @30ºC/min to 300ºC with 4.0 min hold

Approximate run time – 22 minutes
Detector temperature: 330°C

11.2.2. Calibration

NOTE: Refer to SOC-CAL, Calibration of Instruments for Organics Chromatographic Analysis for general calibration procedure, policies, and calculations for various calibration models. Specific calibration procedures are given below:

11.2.2.1. A calibration curve using a minimum of five points is generated using the standards prepared during extraction. The standard level should bracket the expected range of concentrations expected in samples. See section 11.1.3.

11.2.2.2. Calibrate the system prior to conducting any analyses. Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area) versus the concentration in the standard. The ratio of the response to the amount injected, defined as the calibration factor (CF), is calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than 10% over the working range, linearity through the origin can be assumed, and the average calibration factor may be used in place of a calibration curve.

11.2.2.3. If %RSD exceeds 10%, the analyst may plot a linear or quadratic regression curve. Refer to the SOP SOC-CAL for procedures for evaluating alternative curve fits.

11.2.2.4. The calibration is verified by an independent source with each new stock solution. This is done by preparing an independent calibration verification standard (ICV), a dilution of a stock solution purchased from a different vendor, or from a stock solution which is different from the stock used to prepare calibration standards, every time a new stock solution is used. The ICV must meet the same criteria as for CCVs (following section). This is also known as the quality control (QC) reference sample.

11.2.3. Continuing Calibration Verification

11.2.3.1. A continuing calibration standard is analyzed at the beginning of each analytical sequence, and also at the end of each period of continuous instrument operation, or 12 hours, whichever is less. The CCV concentration (in the final extract solution) is 1.25 ug/L. Calculate the % difference (%D) or % drift for the analytes in the CCV using either the calculated concentration or calibration factor. The %D must be within ± 20%.

11.2.3.2. If the CCV fails the ± 20% criteria, evaluate whether the prior samples can be reported: The samples are considered reportable only if the CCV has exceeded the criteria high (>120%) and there are no hits in the sample. Re-analyze any other samples under valid calibration conditions.

11.2.3.3. If a problem related to the GC system has been determined to be the cause of the failed CCV, perform whatever maintenance is necessary before injecting a CCV or recalibrating and proceeding with sample analysis.

11.2.4. Sample Analysis

11.2.4.1. Analyze the samples using the conditions established prior to calibration. Samples are analyzed in a set referred to as an analysis sequence.

11.2.4.2. Identify the method analytes in the sample chromatogram by comparing the retention time of the suspect peaks to retention times of the calibration standards and the laboratory control standards analyzed using identical conditions. Analytes are tentatively identified in samples when peaks are observed in the RT window; however, the experience of the analyst weighs heavily in the interpretation of all chromatograms.

11.2.4.3. Confirmation of all tentative hits must be made. Injecting the sample extract on two columns with dissimilar phases simultaneously provides confirmation. If the retention time matches on both columns, then the hit for the analyte is considered a confirmed hit.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four matrix deionized water samples are spiked with the LCS spike solution, then prepared and analyzed.

12.1.2. For each analyte calculate the mean concentration found in ug/L, and the standard deviation of the four replicates. The mean recovery of each analyte must be between 70-130% of the true value. The RSD must be ≤ 20%. If the results for all analytes meet these criteria, the system performance is acceptable. If any analyte fails to meet the criteria, correct the source of the problem and repeat the test.

12.2. Method Detection Limits and Method Reporting Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank matrix samples with a MDL spiking solution. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Recovery of each analyte in the MDL replicates must be 60-140%. Calculate the MDL for each analyte. Refer to SOP *CE-QA011, Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification*.

12.2.3. The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation, which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the MDL.

12.3. Limits of Detection and Quantification (LOD/LOQ)

12.3.1. The laboratory establishes an LOD for each analyte for each analyte based on the MDL. The MDL and LOD and equivalent unless otherwise specified by project or program requirements (e.g. DOD QSM – see applicable DOD QSM and SOPs).

12.3.2. An LOQ is the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements.

12.3.3. LOD and LOQ verification is performed annually or quarterly depending on accreditation requirements. Refer to the CE-QA011 SOP for details on setting LOD, LOQ, and performing verifications.

12.4. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches (ADM-Batch). In general, these include:

12.4.1. Method Blank

12.4.1.1. A method blank is extracted and analyzed each day to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis.

12.4.2. Lab Control Sample (LCS)

12.4.2.1. The laboratory control sample is composed of analyte-free water into which is spiked a number of appropriate target analytes. The LCS is designed to monitor the accuracy of the procedure. Extract the LCS as in Section 11.

12.4.2.2. A lab control sample (LCS) must be prepared and analyzed with every batch of 10 (or fewer) samples. Calculate the LCS recovery as follows:.

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.3. The acceptance criteria are 60-140%. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.4.4. Matrix Spike

12.4.4.1. A matrix spike (MS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Prepare the MS such that the final concentration of the extract will be 4.375 ug/L. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} x\, 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.4.2. Calculate Relative Percent Difference (RPD) as:

$$\% RPD = \frac{|R1 - R2|}{(R1 + R2)/2} x\, 100$$

Where R1= Higher Result
R2= Lower Result

12.4.4.3. The acceptance limits for the MS are 60-140%. If the MS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

Note: For DOD projects, each batch of samples must contain an associated MS and MSD. If adequate sample for the MS is not available, it must be noted in the case narrative.

12.4.5. Prior to preparation of samples, blanks should be analyzed to determine possible interferences from sample handling steps, reagents, or glassware. If the blanks show contamination, the source of the contamination should be isolated and minimized.

12.4.6. Control charts should be maintained for QC results. The charts should be reviewed periodically for trends in results. Control limits for QC analyses may be determined using the control charts or similar mechanism on an annual basis.

13. DATA REDUCTION AND REPORTING

13.1. The concentration of the analyte(s) in the sample extract (Cex) is calculated using a calibration factor or calibration curve. The concentration of analytes in the original samples is computed using the following equations:

$$Concentration\,(\mu g/L) = \frac{(Cex)(Vf)(D)}{(Vs)}$$

Where Cex = Concentration in extract in µg/L
Vf = Final volume of extract in L
D = Dilution factor
Vs = Volume of sample extracted, L

13.1.1. Sample concentrations are reported when all QC criteria for the analysis has been met. Reported results not meeting QC criteria must be qualified with a standard ALS footnote.

13.2. Reporting

13.2.1. Refer to ADM-RG, Data Reporting and Report Generation for reporting guidelines.

13.2.2. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information and Enviroquant data. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.2.3. As an alternative, reports are generated using Excel© templates on the R: drive. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates.

13.3. Data Review and Assessment

13.3.1. Following primary data interpretation and calculations, a secondary analyst reviews all data. Following generation of the report, the report is also reviewed. Refer to ADM-DREV, Laboratory Data Review Process for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Nonconformity and Corrective Action* (CE-QA008) for corrective action procedures. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example. Table 4 lists typical actions taken.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available. In addition, this procedure was validated through single laboratory studies of accuracy and precision. as specified in Section 12.1.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS EH&S Manual.

16.2. It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

16.3. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17. TRAINING

17.1. Training outline – Training Plan

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of time. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the SOP *for Documentation of Training*.

17.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

19.1. *1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane (DBCP) by Microextraction and Gas Chromatography*, EPA Method 8011, Revision 0, 1992, U.S. Environmental Protection Agency. Environmental Monitoring Systems Laboratory, Cincinnati, Ohio 45268..

19.2. Determinative Chromatographic Separations, EPA SW-846, 8000C March 2003.

20. CHANGES SINCE THE LAST REVISION

20.1. Section 3.5: Removed the word "solid".
20.2. Section 11.1.2: Removed "and working solution" from the first sentence.
20.3. Section 11.1.3: Fixed the header location in the table.
20.4. Section 12.1.1: Changed matrix sand to deionized water.
20.5. Section 12.1.2: Changed ug/Kg to ug/L.
20.6. Updated QA Manager – signature page.

TABLE 1

TARGET COMPOUNDS, MRLs, and MDLs

Analyte	Method Detection Limit (ug/L)	Method Reporting Limit (ug/L)
1,2-Dibromoethane	0.0030	0.01
1,2-Dibromo-3-chloropropane	0.0036	0.01

TABLE 2

Summary of Corrective Actions				
Method Reference	**Control**	**Specification and Frequency**	**Acceptance Criteria**	**Corrective Action**
EPA 8011	ICAL	Prior to sample analysis	% RSD ≤ 10 R2 ≥ 0.995 COD ≥ 0.990	Correct problem then repeat ICAL
EPA 8011	ICV	After ICAL	± 20% Diff	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
EPA 8011	CCV	Prior to sample analysis and end of sequence or 12 hours	± 20% Diff	Correct problem then repeat CCV or repeat ICAL
EPA 8011	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
EPA 8011	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	60-140%	If exceeds limits, re-extract and re-analyze
EPA 8011	Matrix Spike	Include with each analysis batch (up to 20 samples)	60-140%	Evaluate data to determine if the there is a matrix effect or analytical error
EPA 8011	Matrix Spike Duplicates (DOD)	Include with each analysis batch (up to 20 samples)	RPD ≤ 30	Re-homogenize and re-analyze if result is > 5 X the MRL

ALS Environmental

980.0 Pesticide PCB

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980.0 PESTICIDE PCB

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Approved By: __________________Glen Perry__________ Date: _________8/13/2018_
QA Manager – Glen Perry

Approved By: ___________________Rick Bagan________ Date: _______8/13/2018__
Laboratory Director – Rick Bagan

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Organochlorine Pesticides and Polychlorinated Biphenyls by GC/ECD

1.0 Purpose

1.1 To outline the procedure used to extract and analyze for organochlorine pesticides (OCP) and polychlorinated biphenyls (PCB) in environmental samples of solid or liquid matrix.

2.0 References

2.1 Test Methods for Evaluating Solid Waste, USEPA-EMSL, SW-846

2.1.1 Method 8081B: Organochlorine Pesticides by GC

2.1.2 Method 8082: Polychlorinated Biphenyls by GC

2.1.3 Method 3510C: Separatory Funnel Liquid-Liquid Extraction

2.1.4 Method 3550C: Ultrasonic Extraction

2.2 HP ChemStation user manual.

3.0 Definitions & Associated SOPs

3.1 ALSEV Quality Assurance Manual, Revision 9, June 6, 2017.

3.2 ALSEV SOP No. ALSEV 610.0 - Control Charting of Data

3.3 Extraction batch - A group of up to 20 samples extracted within one work day. A Method Blank (MB) and a Blank Spike/Blank Spike Duplicate pair (BS/BSD) is included for each day on which sample(s) are extracted.

3.4 If there is sufficient sample available, a sample MS/MSD will also be extracted.

4.0 Apparatus and Materials

4.1 GC/ECD analytical system

- Hewlett-Packard (HP) 7890 gas chromatograph with dual columns and dual injectors
- HP 7693 Autosampler
- HP G2397A Electron Capture Detectors
- HP Chemstation data system
- Suggested GC columns:

  Restek CLP-1 30m x 0.53mm x 0.5µm

  Restek CLP-2 30m x 0.53mm x 0.42µm

4.2 Sample preparation equipment

- Deionized water system - Barnstead Nanopure Model D4744
- Analytical balances -
  1) Mettler College 2440 Delta Range. (accurate to 0.01 g)
  2) A&D ER-180A. (accurate to 0.0001 g)
- Gas-tight syringes (10, 25, 50, 100, 250, 500, and 1000 µL)
- Graduated cylinder (1000 mL)
- Separatory funnels (2000 mL)
- Erlenmeyer flasks (250 mL)
- Glass beakers (150, 250, and 400 mL)
- Glass filter funnels (100 mm top O.D.)

- K-D concentrators w/10 mL graduated tubes (250 mL)

5.0 Reagents

5.1 Deionized (DI) water: Drawn from Barnstead Nanopure water system.

5.2 Solvents:

- Dichloromethane - high purity grade
- Acetone - high purity grade
- Hexane - high purity grade

5.3 Sodium sulfate - anhydrous

5.4 Silica gel - anhydrous

5.5 Concentrated Sulfuric acid (H_2SO_4)

5.6 18N Sulfuric acid (H_2SO_4) - prepared by diluting concentrated sulfuric acid 1:1 with DI water.

5.7 10M Sodium hydroxide (NaOH) - prepared by dissolving 200g of NaOH into 500 mL of DI water.

5.8 Stock Standards:

5.8.1 Degradation Check solution (DDT/Endrin mix @ 1,2 µg/mL each in isooctane)

5.8.2 Surrogate solution (2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl @ 200ug/mL each in Acetone)

5.8.3 Matrix spike solutions

- Organochlorine Pesticides (2000ug/mL in 1:1 hexane/toluene)
- PCBs (Aroclor 1016/1260 Mix @ 1000 µg/mL in Hexane)

- Chlordane (technical) (2000 µg/mL in Methanol)

5.8.4 Calibration standard solutions:

- Organochlorine Pesticides (200 µg/mL in hexane/toluene 1:1)
- Aroclor 1016/1260 Mix (1000 µg/mL in Isooctane)
- Individual Aroclors: 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268 (100 µg/mL each in Hexane)
- Chlordane (technical) (100 µg/mL in Methanol)
- Toxaphene (100 µg/mL in Hexane)

5.9 Working Standards

5.9.1 Surrogates:

- Soil (200 µg/mL) - use undiluted stock standard.
- Water (20 µg/mL) - dilute 180µL stock in 1.8 mL Hexane.

5.9.2 Degradation Check (0.5 µg/mL) - dilute 250 µL stock plus 1 µL Surrogate stock in 1 mL Hexane.

5.9.3 Calibration Check Standards:

- Organochlorine Pesticides (0.1 µg/mL) - dilute 5 µL stock plus 25 µL Surrogate stock in 10 mL Hexane.
- Organochlorine Pesticides (0.5 µg/mL) - dilute 25 µL stock plus 25 µL Surrogate stock in 10 mL Hexane.
- PCBs (0.5 µg/mL) - dilute 5 µL stock plus 25 µL Surrogate stock in 10 mL Hexane.

- Individual Aroclors (concentration to be determined) - dilute the volume of stock needed for the desired concentration in 1 mL Hexane along with 25 µL of the Water surrogate.

- Chlordane (technical) (0.2 µg/mL) - dilute 20 µL stock plus 25 µL Surrogate stock in 10 mL Hexane.

- Toxaphene (1 µg/mL) - dilute 100 µL stock plus 25 µL Surrogate stock in 10 mL Hexane.

5.9.4 Matrix spike solutions:

- Pesticide Soil Spike (20 – 50 µg/mL) - use undiluted stock standard.

- Pesticide Water Spike (2.0 – 5.0 µg/mL) - dilute 180µL stock in 1.8 mL Hexane.

- PCB Soil Spike (200 µg/mL) - dilute 360 µL stock in 1.8 mL Hexane.

- PCB Water Spike (20 µg/mL) - dilute 36 µL stock in 1.8 mL Hexane.

6.0 Sample Handling and Preservation

6.1 Collection

- Water samples are collected in 1L amber bottles.

- Soil/solid samples are collected in 4oz. jars.

- Oil samples are normally collected in 20 mL Scintillation vials.

- Wipes are taken with Hexane-saturated gauze pads placed in 40 mL VOA vials.

6.2 Samples are stored at 2-6C and should be extracted within 14 days of collection (7 days for waters)

6.3 Sample extracts should be analyzed within 28 days of extraction.

7.0 Procedure

7.1 Extraction.

7.1.1 Water samples:

- For the Method Blank, measure 1000 mL of DI water in a graduated cylinder, and transfer to a 2 L separatory funnel. Repeat twice for the BS/BSD, if required.

- Measure the contents of the sample bottle and transfer to a separatory funnel. Record the actual sample volume in the sample prep log. Check the sample pH. If necessary, use the NaOH and H_2SO_4 solutions to adjust the pH to 6.5-7. (Note: pH adjustment is unnecessary for PCB-only analysis.)

- Add 10 µL of the water surrogate solution (20 µg/mL) to each sample and blank. Add 25 µL of the appropriate spiking solution to BS/BSD.

- Add 50 mL of Dichloromethane to each separatory funnel and shake vigorously for 2 minutes. Allow the solvent to separate from the water and drain into a 250 mL Erlenmeyer flask (one flask for each sample). Repeat twice for 2 minutes and 1 minute respectively.

- While swirling the Erlenmeyer flask, add enough sodium sulfate to take up any residual water in the extract. Pour the contents of the flask into a 250 mL K-D set-up through a filter funnel containing about 30 g of Sodium sulfate. Once fully drained, rinse the funnel with adequate DCM and allow to drain again. Proceed to the concentration step (sec. 7.2).

7.1.2 Soil samples:

- For the Method Blank add about 30 g of Sodium sulfate to a 250 mL beaker. Repeat twice for the BS/BSD, if required.

- Weigh about 25 g of sample into a tared 250 mL beaker. Add about 30 g of Sodium sulfate and mix well.

- Add 25 µL of the soil surrogate solution (200 µg/mL) to each sample and blank immediately before adding 50 mL of 1:1 Acetone/Dichloromethane. For BS/BSD/MS/MSD samples, add 25 µL of the appropriate spiking solution after adding the solvent.

- Sonicate for 3 minutes. Decant the solvent into a 250 mL K-D set-up through a filter funnel containing about 30 g of Sodium sulfate. Use a DCM squirt bottle to rinse the filter after decanting. Repeat twice and allow the filter to drain completely. Once fully drained, rinse the funnel with adequate DCM and allow to drain again. Proceed to the concentration step (sec. 7.2).

7.1.3 Oil samples (for PCBs):

- For the Method Blank add 10 mL of Hexane to a 20 mL scintillation vial. Repeat for the BS/BSD.

- Weigh 1.00 g of the oil sample into a tared scintillation vial. Add 10 mL of Hexane to the vial.

- Add 25 µL of the soil surrogate solution (200 µg/mL) to each sample and blank. For BS/BSD add 25 µL of the PCB soil spike solution (200 µg/mL).

- Sonicate the vials for 5 minutes. Proceed to the cleanup step (sec. 7.3).

7.1.4 Wipes (for PCBs):

- For the Method Blank place a clean gauze pad in a 40 mL VOA vial and add 20 mL of Hexane. Repeat for the BS/BSD.

- Add 20 mL of Hexane to each sample VOA vial.

- Add 20 µL of the soil surrogate solution (200 µg/mL) to each sample and blank/BS/BSD. For BS/BSD, add 50 µL of the PCB soil spike solution (200 µg/mL).

- Sonicate the vials for 5 minutes. Proceed to the cleanup step (sec. 7.3).

7.2 Concentration/Hexane Exchange

- Drop a boiling stone into the K-D set-up containing the sample extract. Attach a Snyder column and place the set-up on the hot water bath.

- When the apparent solvent volume is below visible level of the K-D flask add 10 mL of Hexane through the top of the Snyder column. Continue boiling until the apparent solvent volume is reduced to below 10 mL for soil samples. For water samples, reduce to below 5 mL.

- Allow the K-D set-up to cool to room temperature. Remove the Snyder column and K-D flask. For soil extracts bring the volume to 10 mL with hexane and proceed to the cleanup step (sec. 7.3). For water extracts, bring the final volume to 5 mL with hexane and proceed to the cleanup step (sec. 7.3).

7.3 Silica Gel Cleanup

7.3.1 Silica Gel-Acid (for PCBs only) - to be used for extracts containing high levels of interfering hydrocarbons

10 mL extracts: Add about 0.5 mL concentrated sulfuric acid to a scintillation vial. Pour the extract into the scintillation vial and shake. Then add a heaping teaspoon of silica gel. Shake the capped vial vigorously and centrifuge for about a minute. Repeat this process until solution is clear. Draw off the solvent using a Pasteur pipette, being **very careful not to disturb the acid layer**. Transfer the extract to two autosampler vials.
Note: The process may be repeated, if necessary, by decanting the solvent into a fresh scintillation vial. Use proportionally less acid and silica gel as the extract volume is reduced.

5 mL extracts: Same procedure as the 10 mL extracts but use about half as much sulfuric acid and silica gel.

7.3.2 Copper - to be used for samples containing elemental sulfur.

Add a pinch of granulated copper to the autosampler vial containing the extract. Shake the capped vial vigorously for several minutes and allow any precipitate to settle out.

7.4 Analysis

7.4.1 Calibration

- Prepare initial calibration standards at a minimum of 5 levels covering the linear range of the detector. (Include the surrogates 0.05 µg/mL to 1.0 µg/mL in the standards)
    - Pesticides: 0.01 µg/mL to 0.5 µg/mL
    - PCBs: 0.1 µg/mL to 5.0 µg/mL
    - Chlordane (technical): 0.02 µg/mL to 2.0 µg/mL
    - Toxaphene: 0.1 µg/mL to 10.0 µg/mL

- For pesticides a degradation check must be performed before proceeding with the calibration. The breakdown of DDT and Endrin from the 0.5 µg/mL standard must be no greater than 15% for each compound.

- For PCBs, Chlordane (technical), and Toxaphene 5 peaks are chosen to represent the entire mixture (in the case of PCBs: 5 each for 1016 and 1260). Ideally, choose 5 peaks that encompass the full retention time spectrum of the mixture.

- The calibration curves must have a correlation coefficient of 0.99 or greater.

7.4.2 Sample analysis

- Begin the analytical sequence with a primer (~ 20 µg/mL Pesticide standard) followed by a solvent blank. If analyzing for pesticides, verify acceptable breakdown with a degradation check before running calibration check (CCV) standards.

- The CCV results must be within 20% of the standard concentration. For PCBs, calculate the average of all calibrated peaks in the CCV. If the criterion is met, continue the sequence with the method blanks, spikes, and samples.

- CCVs should be run after every 10 samples and at the end of the sequence. (For pesticides alternate between the 0.01 µg/mL and 0.5 µg/mL standards.)

7.4.3 Quantitation

- Use the data available from both columns to make positive identifications of compounds and/or mixtures. Overlay and mirror-image comparison to standards is particularly useful for identifying Aroclors.

- If a sample contains an Aroclor, estimate the concentration based on the CCV and prepare a calibration standard containing approximately that concentration of the Aroclor and 0.5 µg/mL of the surrogates. (If the sample concentration appears to be above the linear range of the detector, run a dilution of the extract along with a standard near the resulting concentration.) The standard must be injected within 72 hours of the sample in order to serve as a single-point calibration for the sample. As with the 1016/1260 calibration, choose 5 peaks to represent the mixture. (If the identified Aroclor is 1016 or 1260, the multi-point curves may be used, provided that the concentration is within the calibration range.)

- Calculate the sample concentration as follows:

    - Water: $\mu g/L_{sample} = \mu g/mL_{extract} \times \frac{mL_{extract}}{L_{sample}}$
    - Soil or oil: $mg/kg_{sample} = \mu g/mL_{extract} \times \frac{mL_{extract}}{g_{sample}}$
    - Wipe: $\mu g/wipe_{sample} = \mu g/mL_{extract} \times \frac{mL_{extract}}{wipe_{sample}}$

8.0 Quality Control

8.1 Instrument QC

8.1.1 Control Charts - Surrogate and Spike Control limits should be determined for soils and waters for Pesticides and PCBs. Refer to SOP No. ALSEV 610.0.

8.1.2 Method detection limits - Detection limits should be determined for soils and waters for Pesticides and PCBs. For general guidance, refer to Chapter 1, Section 5.0 of SW-846, Revision 7, June 2014. Practical Quantitation Limits (PQLs) are set as 3 times the MDLs.

8.1.3 Degradation check & Calibration verification - A Degradation check is performed at the beginning of any sequence containing samples for pesticide analysis. CCV standards for each analyte of interest are included to bracket each group of up to 10 samples in a sequence. See sec. 7.3 for acceptance criteria.

8.2 Batch QC

8.2.1 Method Blank - A blank sample, carried through the same steps as the actual sample(s) should be included with each extraction batch on each day for which sample(s) are extracted. It should be free of target analytes at the PQLs specified in the method.

8.2.2 Blank Spike/Blank Spike Duplicate - A BS/BSD pair, carried through the same steps as the actual sample(s) should also be included in each extraction batch on each day for which sample(s) are extracted. The percent recoveries and RPD should fall within the control limits established for the analysis.

8.2.3 Matrix Spike/Matrix Spike Duplicate - An MS/MSD (when sufficient sample is provided), carried through the same steps as the actual sample(s) should be included with each extraction batch for which sample(s) are extracted. The percent recoveries should fall within the control limits established for the analysis.

8.3 Surrogate recoveries - Surrogates are added to all samples, blanks, blank spikes, and matrix spikes. Percent recoveries should fall within the control limits established for the analysis.

8.4 Interferences

8.4.1 Background contamination - Method Blanks are used to monitor background contamination. Target analytes detected in the Method Blank may result from cross-contamination during the extraction process, or from carryover in the GC system. When target compounds are detected in a Method Blank, the source of contamination should be determined, and if warranted, associated samples should be reextracted and/or reanalyzed.

8.4.2 Matrix interference - Non-target compounds in the sample extract may interfere with the analysis in various ways. Compounds which coelute with target analytes may make accurate quantitation impossible. In this case the best recourse is to raise the reporting limit for the analyte. Other matrix elements may adversely affect the analysis by adsorption of target analytes in the extraction process, or through interactions within the GC system resulting in poor chromatography, retention time shifts, or diminished response for certain analytes. Careful monitoring of surrogate recoveries and retention times should reveal when this occurs.

8.5 Non-Conformance - When QC objectives fail to be met, and there is no way to correct the deficiency (e.g. reextraction, reanalysis), submit a Non-Conformance Report with the sample data, and place a copy in the lab-wide Non-Conformance database. Refer to the Quality Assurance Manual for further guidance.

8.6 Instrument maintenance

8.6.1 Routine maintenance - The injection port end of the GC system requires relatively frequent attention in order to maintain acceptable performance. When check standards indicate excessive peak tailing, or when the standards fail to meet the acceptance criteria outlined in sec. 7.4, the following corrective measures may be tried:

- Replace the inlet liner with a clean, deactivated one. (Also check for residue on the underside of the inlet weldment, and clean with a cotton swab and solvent if necessary.)
- Remove anywhere from an inch to several loops of column.
- Replace the septum.
- Inspect the gold-plated inlet seal. Clean or replace depending on condition.
- Replace the column.

8.6.2 Detector - Over time the electron capture detector may become contaminated resulting in a high and/or erratic baseline. The following measures may be taken to improve this condition:

- Disconnect the column from the detector and cap the base of the makeup gas adapter. Set the detector to 350C for 1 hour. If the signal decreases, extend the bake period until the signal stabilizes.
- If bakeout does not improve the baseline, and other potential problem sources (e.g. gas impurities, leaks, column bleed) have been eliminated, the detector will likely need to be reconditioned by a certified repair facility.

9.0 Records Management

- Initial calibration records are filed in the initial calibration filing area.
- All sample specific records are submitted in the appropriate project folders along with a summary of the relevant quality control data.
- All instrument specific daily records (e.g. continuing calibrations and degradation checks) are filed in the analytical sequence section of the filing area.
- Sequence logs are printed to provide a record of which samples were run in each sequence.

10.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Using solvents (Methylene chloride, Acetone and Hexane) and adding surrogate (TCMX and DCB) during extraction.	Accidental spills and splashes.	Use PPE (gloves, protective clothing, eye protection). Perform task under fumehood.
Job Task #2:	**Hazards**	**Preventative Measures**
Using hot water bath to boil down extract.	Inhalation of fumes.	Perform task under fumehood. Place sash window down to the maximum protection level.
Job Task #3:	**Hazards**	**Preventative Measures**
Washing and handling glassware.	Skin cuts.	Use PPE. Avoid using chipped/slightly broken glassware.
Job Task #4:	**Hazards**	**Preventative Measures**
Disposal of excess or refuse extract and soil waste.	Inhalation of fumes and Skin contact.	Place under fumehood to dry/evaporate before disposing refuse in an approved labeled container.
Job Task #5:	**Hazards**	**Preventative Measures**
Using Hydrocholric acid and silica gel to clean up extract.	Skin contact.	Use PPE.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

Colorimetric Determination of Hexavalent Chromium (Cr^{+6}) in Soils and Waters

1.0 Purpose

1.1 To outline the procedure for the determination of the concentration of hexavalent chromium (Cr^{+6}) in soil and water samples.

2.0 References

2.1 ALSEV Quality Assurance Manual (QAM).

2.2 Test Methods for Evaluating Solid Waste, Physical / Chemical Methods, SW-846, “Method 7196A: Chromium, Hexavalent (Colorimetric)”, Environmental Protection Agency, Revision 1, July 1992.

2.3 ALSEV SOP # 706.0, pH of Water and Soil, 8/9/1999.

2.4 Thermo Scientific Genesys 20 spectrophotometer, Operator’s Instructions.

3.0 Definitions

3.1 Analytical Batch – The basic unit for quality control. An analytical batch represents samples, which are analyzed together with the same method, same lots of reagents and same steps in common to each sample, with the same time period. The maximum batch size is 20 samples.

3.2 Initial Calibration Curve (ICal) – A minimum of 5 different Cr^{+6} concentrations, ranging from 10 ug/L to 1000 ug/L, made from a stock solution.

3.3 Initial Calibration Verification (ICV) – The first mid-range working standard used to verify that the instrument is functioning correctly and that the initial calibration is still valid. The value obtained for this analysis must not vary from the true value by more than 10%.

3.4 Continuing Calibration Verification (CCV) – Any subsequent mid-range working standard diluted from the stock standard used to verify that the analytical system is operating in a manner comparable to that at the time of initial calibration. The value obtained for this analysis must not vary from the true value by more than 10%.

3.5 Initial Calibration Blank (ICB) – A blank used to verify the calibration curve, that is run immediately before the ICV and must have a value that is less than the detection limit. If the ICB fails, the instrument must be recalibrated. Other corrective actions may also be taken, which may include cleaning the instrument, etc.

3.6 Continuing Calibration Blank (CCB) – A calibration check standard used to verify the calibration curve that is run after every 10 samples and at the end of every sample run. The CCB must have a value that is less than the detection limit. If the CCB fails, all samples up to a preceding acceptable ICB or CCB must be rerun.

3.7 Second Source Standard Solution – A calibration check standard prepared from a source independent of the primary calibration standard. It is used to verify the accuracy of the initial calibration curve.

3.8 Method Blank (MB) – An artificial sample designed to monitor the introduction of artifacts into the analytical scheme. The method blank is taken through each step of the analysis.

3.9 Blank Spike (BS) – A quality control sample prepared by adding a second source standard solution to a blank matrix and carried through the entire analysis process. Results of the blank spike are used to monitor method performance and must fall with 10% of the true value for the analytical batch to be valid.

3.10 Matrix Spike (MS) – A quality control sample prepared by adding a second source standard solution to a sample matrix and carried through the entire analysis process. Results of the matrix spike are used to monitor method performance on actual samples. Recoveries deviating more than 50% from the expected value should be qualified appropriately.

3.11 Sample Duplicate (DUP) – A replicate of a sample used to determine the precision of the analytical method for the sample matrix. Relative percent difference (RPD) exceeding 50% should be qualified appropriately.

3.12 Reporting Limit – The smallest amount of analyte that can be detected and reliably quantified and is based on the lowest standard. For this method, the reporting limit is 5.0 mg/kg Cr^{+6} for soil samples and 10 µg/L Cr^{+6} in water samples.

3.13 Method Detection Limit (MDL) – A number, with units of concentration, generated according to the procedure described in 40 CFR, Part 136, Appendix B. The MDL is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

4.0 Apparatus and Materials

4.1 Analytical Instruments

4.1.1 Ultraviolet / Visible (UV/Vis) Spectrophotometer: Thermo Scientific Genesys 20 with 1 cm quartz cell.

4.1.2 Orion pH probe and processor with automatic temperature compensation.

4.2 Sample Preparation Equipment

4.2.1 Balances

4.2.1.1 Analytical Balance, capable of weighing to 0.1 mg

4.2.1.2 Top loading balance, capable of weighing to 0.01 g

4.2.2 Weighing paper

4.2.3 Weighing pans

4.2.4 100mL volumetric flasks with caps

4.2.5 250 mL volumetric flasks with caps

4.2.6 Variable volume pipettor with range of 0.5mL – 5.0mL

4.2.7 5 mL pipet tips

4.2.8 Variable volume pipettor with range of 10uL – 1.0mL

4.2.9 1 mL pipet tips

4.2.10 47mm diameter filter funnel, capacity 300 mL

4.2.11 47mm diameter membrane filters, pore size 0.45 µm

4.2.12 50mL polypropylene digestion cups

4.2.13 Oven, standard laboratory-type

4.2.14 Miscellaneous glassware typically used in an analytical laboratory such as funnels, spatulas, weighing pans, beakers, volumetric flasks, desiccators, magnetic stirrers, and stir bars

5.0 Reagents

5.1 Deionized water (DI) – Drawn from ELGA PURELAB water system.

5.2 Sulfuric Acid – Concentrated, reagent grade acid that is suitable for trace element analysis and is purchased from vendors.

5.2.1 Sulfuric Acid, 10% (v/v) – 10 mL reagent grade sulfuric acid diluted to 100 ml with DI water.

5.3 Potassium Dichromate – The neat source of Cr^{+6} (from $K_2Cr_2O_7$) standard and second source standard purchased from separate vendors with independent lot numbers, stored at room temperature. Each neat standard must be dried at 103°F for 1-2 hours and then dessicated for 1-2 hours prior to use. The $K_2Cr_2O_7$ should be an analytical reagent grade chemical.

5.3.1 Potassium Dichromate Stock Solution – The Cr^{+6} stock solution is prepared by dissolving 0.3535g $K_2Cr_2O_7$ in 250mL of DI water to give a concentration of 500 mg/L of Cr^{+6}. The stock solution is prepared fresh after one year or sooner if comparison to check standards indicates >15% difference.

5.3.2 Potassium Dichromate Working Standard – The working standard is prepared by diluting 10mL of the Stock Solution into 100mL of DI water to give a concentration of 50 mg/L of Cr^{+6}. The working solution is prepared fresh after one year or sooner if comparison to check standards indicates >15% difference.

5.4 Diphenylcarbazide Solution – A solution of 1,5-diphenylcarbazide dissolved in acetone to give a 0.5% solution. Add 0.250g to a 50 mL volumetric flask and bring to final volume with acetone.

6.0 Sample Collection, Preservation and Handling

6.1 Soil Samples are normally collected in 4 oz wide mouth glass containers with Teflon lines closures. Water samples are collected in 16 oz HDPE bottles.

6.2 Samples are shipped in coolers with coolant and appropriate packaging to prevent cross contamination and breakage.

6.3 Soil samples are to be extracted within 28 days of sampling. Water samples and soil extractions must be analyzed within 24 hours of sampling or extraction.

6.4 All samples and the extracts should be stored at 4° C until analyzed.

7.0 Procedure

7.1 Soil Sample Extraction

7.1.1 Thoroughly mix samples and discard any foreign objects (rocks, twigs, etc). Weigh 1.0 to 12.5 grams of sample into a 250mL HPDE bottle. Weigh an identical amount of one sample in the batch for both a duplicate and a matrix spike. Determine the % solids of each sample, and record the dry weights to the nearest .01g in the Cr^{+6} analysis logbook.

7.1.2 Add 100mL of DI water to each sample, method blank, and blank spike/blank spike duplicate. Add 200 uL Cr^{+6} spiking solution to each of the spikes. Vortex each bottle to mix, and allow at least 1 hour for sediment to settle before centrifuging.

7.1.3 Filter the liquid with a 0.45um filter funnel to obtain at least 60 mL of sample extract.

7.1.4 Sample must be analyzed within 24 hours of this extraction.

7.2 Calibration Standards Preparation

7.2.1 Prepare calibration standards at 10, 50, 100, 500, and 1000ug/L in 50mL digestion cups. Add a volume of $K_2Cr_2O_7$ working standard to DI water and bring to 50 mL total volume. The microliter amount of working standard will be equal to the concentration of the calibration standard.

7.2.2 The calibration curve is verified using a mid-level continuing calibration standard. This standard is analyzed at the beginning and at the end of the analytical sequence and after every 10 samples within the analytical sequence.

7.2.3 Calibration standards and all QC samples are to receive the same color development procedure as samples once the standards have been made to the appropriate concentration at 50mL final volume.

7.3 Sample, Extract and Standard Preparation

7.3.1 The analytical batch consists of 20 samples. The following QC samples must be analyzed with each batch (see sec. 3.0):

1 MB per batch

1 BS/BSD per sample batch

1 DUP per batch

1 MS sample per batch

7.3.2 Pour 50 mL of the sample or extract into digestion cup.

7.3.3 Color Development

7.3.3.1 Add 0.5 mL 10% sulfuric acid to each sample (including standards, blanks, and spikes). After mixing, check each sample to ensure the pH is 2.0 ±0.5.

7.3.3.2 Add 2.0 mL diphenylcarbazide solution and mix.

7.3.3.3 Allow samples to develop for 10 minutes.

7.3.3.4 Samples and standards should be read within 15 minutes once color development is completed.

7.3.4 Reading the Samples and Extracts on the Spectrophotometer

7.3.4.1 For operation, calibration or general use and care of the spectrophotometer, reference Genesys 20 Operator's Manual.

7.3.4.2 Turn on the spectrophotometer, and allow the instrument to warm up for at least 30 minutes prior to usage. Set the wavelength to 540 nm. Press A/T/C until ug/L units appear on the screen

7.3.4.3 Rinse the cell with sample once before each reading. Be careful not to touch the front and rear sides of the cell, and wipe away any water from the outer surface.

7.3.4.3 Zero the meter with prepared blank, and press the Print button twice to print out the zero value. For each calibration standard, use the up/down buttons to step to the correct concentration. When a standard value is set, press Print.

7.3.4.2 After the highest calibration level has been set, read the calibration blank followed by a mid-level (100ug/L) calibration standard. The blank should read below the reporting limit, and the calibration verification should be within 10% of the actual value. Print result of each reading.

7.3.4.3 Read the samples and associated batch QC. Include a blank and calibration standard after every 10 samples and at the end of the analytical sequence. Print result of each reading.

7.3.4.5 Transcribe the spectrophotometer readings to the logbook, and tape the printout to the page.

7.4 Calculations

7.4.1 Moisture / Dry Soil Determination

Record the weight of a pan and tare the balance. Weigh 10 - 20 grams of the sample into the tared pan. Determine percent moisture by drying at least 1 hour at approximately 100°C. The percent dry weight is calculated as:

% dry soil = (C-B) / A x 100

Where:
A = wet sample weight
B = weight of pan
C = weight of pan + dry sample

7.4.2 Cr^{+6} Determination

7.4.2.1 Use the spectrophotometer readings to calculate the sample results. The example calculations are:

Soil Sample Results: (mg/kg) = [(A x B) / C] x D
or
Water sample Results: (ug/L) = A x D

Where:
A = Solution concentration (ug/L)
B = Total extract volume (L)
C = Dry weight of sample (g)
D = Dilution factor

7.4.2.2 Soil Sample Results < 5 mg/kg shall be reported as ND(<5 mg/kg).

7.4.2.3 Water Sample Results <10 ug/L shall be reported as ND(<10 ug/L).

8.0 Quality Control

8.1 On-going quality control

8.1.1 Quality control acceptance criteria are given in Appendix 1.

8.1.2 Extract a method blank per section 7.3.1.

8.1.2.1 The method blank must show a non-detect for Cr^{+6} and is recorded as Cr^{+6} < 5.0 mg/Kg for soils or <0.5mg/L for waters. If the method blank fails to meet acceptance criteria, then diagnose the problem and take corrective action.

8.1.2.2 Analyze the method blank sample for the analytical batch prior to the duplicates and field samples.

8.1.3 Extract a duplicate per section 7.3.1.

8.1.4 Calculate the relative percent difference (RPD) for duplicate analyses using the following equation, where D_1 and D_2 represent the results from duplicate analyses:

$$RPD = D_1 - D_2 / (D_1 + D_2) / 2 \text{ X } 100$$

Compare the RPD with the current acceptance criteria for this procedure. If the RPD meets the acceptance criteria, all the samples in the analytical batch are acceptable. If the RPD fails to meet criteria, diagnose the problem and discuss with laboratory director or QC Officer to determine in the analytical batch is to be reported.

8.1.5 Method Detection Limit Determination

8.1.5.1 A method detection limit determination is performed using the procedure described in 40 CFR, Part 36m Appendix B.

8.1.5.2 The method detection limit determination is to be performed at least once to demonstrate confidence levels. Project specific plans may require additional determinations at specified frequencies.

8.2 Nonconformance and Corrective Action

8.2.1 Any discrepancy affecting the quality of the data for any sample is documented on a nonconformance memo (NCM) ncar or within the project file.

9.0 Records Management

9.1 Sample results and the QC results are maintained in bound notebook.

9.2 After an independent data review has been completed, a copy of the pertinent sample data from the bound notebook is filed in the appropriate client project files.

Color blank readings

Colorimetric Determination of Cr^{+6} in Soils, Appendix 1
Acceptance Criteria for Quality Control

	% Recovery	Relative % Difference
Calibration Verification	90-110	
Blank Spike and Duplicate	85-115	25
Sample Duplicate		25
Matrix Spikes	85-115	

820.0 ICPMS Metals

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820.0 ICPMS METALS

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Laboratory Director – Rick Bagan

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Analysis of Metals by ICP-MS

1.0 Purpose

1.1 To outline the procedure for the determination of trace elements in aqueous and solid samples by ICP-MS.

2.0 References

2.1 EPA Method 200.8 – Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry, revision 5.4, 1994

2.2 EPA SW-846 Method 6020B– Inductively Coupled Plasma-Mass Spectrometry, revision 2, July 2014

2.3 EPA SW-846 Method 3010A – Acid Digestion of Aqueous Samples, revision 1, July 1992

2.4 EPA SW-846 Method 3050B – Acid Digestion of Sediments, Sludges and Soils, revision 2, December 1996

2.5 ALS Everett SOP 801.0 – ICPMS Water Digestion

2.6 ALS Everett SOP 802.0 – ICPMS Soil Digestion

2.7 ALS Everett Quality Assurance Manual (ALSEV QAM)

2.8 Agilent 7800 ICP-MS Hardware and MassHunter User Manuals

3.0 Definitions

3.1 Analytical Batch - A group of up to 20 samples digested on the same day. The batch includes a Method Blank, Blank Spike/Blank Spike Duplicate pair, and a Matrix Spike/Matrix Spike Duplicate pair.

3.2 Method Detection Limit (MDL) – Theoretical low concentration limit determined according to 40 CFR, Part 136 Appendix B. The MDL should be determined annually at a minimum.

3.3 Practical Quantitation Limit (PQL) – The smallest amount of analyte that can be reliably quantified. For this method the PQL is 3 times the MDL.

3.4 Determination of Linear Range (LDR) – The highest concentration level for which an element is recovered to within 10% of the expected value. A series of standards should be run at least quarterly to make this determination for all elements.

4.0 Apparatus and Materials

4.1 Agilent 7800 ICP-MS with Octopole Reaction System (Helium mode)

4.2 ISIS 3 sample introduction system

4.3 Agilent SPS 4 autosampler

4.4 Autopipettors (0.01mL – 0.1mL, 0.1mL – 1mL, 0.5mL – 5mL, 1mL – 10mL) with disposable tips.

4.5 50 mL volumetric flasks (class A)

4.6 50 mL polypropylene self-standing conical bottom centrifuge tubes

4.7 50 mL polypropylene digestion cups with screw caps, Environmental Express

4.8 FilterMate filtration assemblies, Environmental Express

4.7 17 x 100 mm polypropylene culture tubes

5.0 Reagents and Standards

5.1 Deionized water, ELGA Purelab Flex water (resistance ≥ 17 MΩ).

5.2 Concentrated Nitric Acid, Baker instra-analyzed or equivalent.

5.3 Concentrated Hydrochloric Acid, Baker instra-analyzed or equivalent.

5.4 Stock standards - purchased as certified solutions. (See Table 1)

5.4.1 Tuning solution – 10 ug/mL, SPEX

5.4.2 Internal Standard solution – 10 ug/mL, VHG Labs

5.4.3 Germanium (ISTD) – 1000 ug/mL, SPEX

5.4.4 ALS Custom Calibration Mix – 10 or 1000 ug/mL, Inorganic Ventures

5.4.5 Aluminum – 1000 ug/mL, Inorganic Ventures

5.4.6 Strontium – 1000 ug/mL, Inorganic Ventures

5.4.7 Tin – 1000 ug/mL, Inorganic Ventures

5.4.8 Calibration Mix 1 (2nd source) – 10 ug/mL, SPEX

5.4.9 Calibration Mix 3 (2nd source) – 1000 ug/mL, SPEX

5.4.10 Calibration Mix 5 (2nd source) – 10 ug/mL, SPEX

5.4.11 Aluminum (2nd source) – 1000 ug/mL, SPEX

5.5 Working standards (see Table 2)

5.5.1 ICPMS Calibration Standards – Prepared at levels of 0.2 to 200 ppb (20 to 20,000 ppb for Al, Ca, Fe, K, Mg, Na)

5.5.2 ICV – Initial Calibration Verification standard at the midpoint of the calibration. It is prepared fresh daily from a source other than that used to prepare the calibration standards.

5.5.3 CCV – Continuing Calibration Verification standard. It is prepared at the same time and from the same source as the calibration standards.

5.5.4 LLCCVs – Low-level Calibration Verification standards at the 3 lowest levels of the calibration. These are prepared at the same time and from the same source as the calibration standards.

5.5.5 ICSA – Interference Check Solution A. Primarily, this is used to assess the potential for false positives due to matrix interferences. It is prepared at the same time as the calibration standards.

5.5.6 ICSAB – Interference Check Solution AB. (ICSA and CCV combined)

Primarily, this is used to assess matrix effect on viability of the calibration. It is prepared at the same time as the calibration standards.

5.5.7 Tuning Solution – used to tune the instrument and to assess instrument performance.

5.5.8 ISTD solution – used to normalize data and to monitor instrument performance and matrix effects for each run.

5.5.9 Spiking solution – A standard solution added to samples prior to digestion.

5.6 Liquid Argon, high purity

5.7 Helium, ultra-high purity

6.0 Sample Handling and Preservation

6.1 Aqueous samples for metals analysis are collected in 500 mL HDPE bottles. Soil samples are collected in 4 or 8 ounce glass jars.

6.2 Aqueous samples for total metals must be preserved with nitric acid at the time of collection. Aqueous samples for dissolved metals analysis should be filtered at the time of collection or as soon as possible prior to preservation.

6.4 All samples should be analyzed within 6 months of collection and stored 4°C.

7.0 Procedure

7.1 Sample preparation

7.1.1 Digestion – refer to SOPs 801.0 and 802.0. Digests are contained in 50 mL digestion cups and are transferred as dilutions to 17 x 100 mm culture tubes for analysis.

7.1.2 Water digests are diluted 2.5-fold prior to analysis, resulting in a net 1.25x dilution. If the diluted digest contains significant suspended solids, it should be filtered with a FilterMate assembly. Samples suspected of containing very high levels of minerals or other elements of interest should be diluted further before introduction to ICPMS.

7.1.3 Soil digests containing significant suspended solids should be filtered with a FilterMate assembly. All digests are diluted 10-fold prior to analysis. Samples suspected of containing very high levels of elements of interest should be diluted further before introduction to ICPMS.

7.1.4 Post-digestion spikes are performed after filtration and dilution. Add ICPMS spiking solution to an aliquot of the digest of the sample that was used for MS/MSD. The amount added should make the expected concentration the same as that of the Matrix Spikes.

7.1.5 Make an additional 1:5 dilution digest of the sample that was used for MS/MSD to serve as a Dilution Test.

7.2 Instrument Startup and Batch Configuration

7.2.1 ICPMS Acquisition Parameters – See Table 3

7.2.2 Empty waste containers if necessary. Turn on water chiller. Clamp tubes in place on the peristaltic pump. Remove caps from calibration tubes and rinse bottles. (Refill if necessary)

7.2.2 Ignite the plasma, initiating instrument startup routine. This consists of warmup, autotune, and performance check. (The entire process takes about 30 minutes.)

7.2.3 As startup routine proceeds, select New Batch Folder from the File drop-down menu. Ordinarily, create a new batch from the most recent batch. The typical sequence flow consists of a Calibration block followed by QC

Check block followed by a Sample block. A Periodic block consisting of CCV and CCB will be spliced in automatically every 10 runs following the ICV.

7.2.4 Edit the Sample block to contain the new digestion batch information. When finished click Validate Method. If no errors are found, the Batch may be added to the Queue.

7.2.5 Once the Batch is added to the Queue, click Pause at End. This will facilitate adding new samples or reruns to the sequence, and make it possible to export a copy of the Batch Log to an Excel file without having to requeue the batch.

7.3 Performance Report and Tune Evaluation

7.3.1 Check the Performance Report for the following:

- Oxide ratio (m/z 156/140) < 5%.
- Doubly-charged ratio (m/z 69/138) < 5%
- Change in sensitivity compared to recent performance

7.3.2 Check the Tune Report to ensure the following criteria are met for m/z 7, 89, 205 in five replicate analyses of the Tuning solution:

- RSD less than 5%
- mass axis within ± 0.1 amu of nominal
- peak width at 10% of peak height within 0.65-0.8 amu

7.3.2 Failure to meet the above criteria usually calls for some degree of instrument maintenance. Routine maintenance (in order of complexity) is as follows:

- Replace peristaltic pump tubing
- Clean or replace Sampler and Skimmer cones
- Clean Torch, Bonnet, Spray chamber, and MicroMist Nebulizer
- Clean or replace Lens stack

7.3.4 Record all maintenance performed in the instrument maintenance log. Use the format of: Date, Analyst Initials, Brief narrative describing problem, action taken, and result.

7.4 Calibration and initial QC checks

7.4.1 Interference Equations – See Table 4

7.4.2 The instrument is calibrated at the beginning of an analytical batch and as

necessary thereafter should calibration or internal standard checks fail to meet method criteria. The calibration consists of a Blank followed by seven levels (0.2, 0.5, 2, 5, 10, 100, and 200 ppb*.) See Table 2 for preparation instructions.
* Levels are 100 times higher for Al, Ca, Fe, K, Mg, and Na

7.4.3 Once the calibration is complete, check the curves to ensure the minimum correlation coefficient of 0.995 is met for all elements. High points may be dropped in order to achieve required linearity. (This will limit the quantitation range however)

7.4.4 The ICV is run after the calibration. All elements of interest must be within ± 10% of the expected value.

7.4.5 The ICB is run after the ICV. All elements of interest must be less than ½ of the reporting limit.

7.4.6 LLCCVs at 0.2/20, 0.5/50, and 2/200 ppb are run following the ICB. If an element is to be reported down to these levels, it must be within ± 20% of the expected value.

7.4.7 The ICSA is run after the LLCCVs. All elements of interest not in the standard must be less than ½ of the reporting limit. All elements of interest contained in the standard must be within ± 20% of the expected value.

7.4.8 The ICSAB is run after the ICSA. All elements of interest must be within ± 20% of the expected value.

7.5 Sample analysis and batch QC

7.5.4 A CCV and CCB are run at the beginning of the Sample block and every 10 runs thereafter. For the CCV all elements of interest must be within ± 10% of the expected value. For the CCB all elements of interest must be less than ½ of the reporting limit.

7.5.5 Internal Standard counts must be within 70 – 125% of the reference (ICAL Blank) for batch samples. For instrument QC checks the acceptance range is 80 – 120%.

7.5.6 The Method Blank should not have elements of interest higher than 10% of the level reported for a sample, or higher than 2.2 x MDL (whichever is greater.)

7.5.7 The BS and BSD recoveries should be 85 – 115%, and the RPD should be no more than 20%.

7.5.8 The MS and MSD recoveries should be 75 – 125%, and the RPD should be no more than 20%.

7.5.9 The Post-digestion Spike recovery should be 80 – 120%

7.5.10 The 1:5 Dilution Test should agree with the original determination to within 10% for any element with concentration within the linear range and at least 25 times the reporting limit.

7.5.11 Samples with concentrations of elements of interest that exceed the upper limit determined by the LDR (sec. 3.4) should be diluted and reanalyzed. (The upper limit becomes the highest calibration point if any levels are dropped.)

7.5.12 Silver solubility issues make it a special case. Any water sample with an apparent final digest concentration above 100 µg/L must be diluted prior to digestion. Redigest samples if necessary.

7.6 QC failures and corrective action (see Table 5)

7.6.1 Instrument QC – Failures include calibration checks outside of allowable range, detection of elements about the allowable limit for calibration blanks, and internal standard counts out of allowable range, but not due to matrix interference. Corrective actions generally involve maintenance and/or recalibration.

7.6.2 Batch QC - Failures include spike recoveries outside of allowable range, detection of elements about the allowable limit for method blanks, and internal standard counts out of allowable range due to matrix interference. Corrective actions range from simply reanalyzing a sample at a dilution to redigesting an entire batch.

8.0 Data analysis and reporting

8.1 Calculations

8.1.1 Final concentration is calculated as follows:

Soil: $IC \times (V/W) \times Dm \times Da = \text{mg/kg Hg}$

Water: $IC \times Dm \times Da = \text{ug/L Hg}$

where IC = instrument concentration (ug/L),

V = nominal digest final volume (0.05 L),

W = amount of soil (dry wt.) in grams,

Dm = method dilution (1.25 for water, 10 for soil)

Da = additional dilution

8.1.2 Percent recovery and Relative percent difference are calculated as follows:

Blank Spike: % Recovery = (Spike result/Expected spike result) x 100

$$\text{Matrix Spike: \% Recovery} = \frac{(\text{Spike result} - \text{Sample result}) \times 100}{\text{Expected spike result}}$$

$$\text{RPD} = \frac{(\text{Spike Recovery} - \text{Spike Duplicate Recovery}) \times 100}{(\text{Spike Recovery} + \text{Spike Duplicate Recovery})/2}$$

8.2 Print Hardcopy reports for all samples and batch QC. Use a highlighter to indicate the elements and particular isotopes to be reported for a given sample.

8.3 Select sample(s) in the Data Analysis batch table, click the Report drop-down menu, and choose LIMS > Export selected samples. This will create a csv file from which the data may be parsed for upload to the LIMS. (Note: before exporting, the current limsexport.csv file should be deleted from the destination folder)

8.4 Open the parser, make any desired changes to the isotope selection table, select the limsexport.csv file, and click the Parse Data button. Click Review Data to verify the correct data files have been parsed. For soils, print a copy of the report for each sample so that the data reviewer has a hardcopy of results converted to soil units. Finally, click Export Parsed Data to create the csv file for LIMS upload.

8.5 Enter the raw data results for the batch QC samples in an Excel template to create a coversheet for the batch. The coversheet will include the results of the MB, BSD/BSD, MS/MSD, Post-digestion spike, and Dilution test for all elements of interest in the batch. Print a copy to be included with the raw data for each work order in the batch.

9.0 Records Management

9.1 ICPMS data is stored electronically by batch. Each batch includes the instrument tuning parameters along with the raw data.

9.2 Hardcopy reports of raw data for all analytical batch QC (calibration and interference checks) along with a copy of the batch sequence log are saved in a batch file folder for archiving.

9.3 Hardcopy reports of raw sample data are submitted along with associated QC summaries for peer review.

10.0 Safety

10.1 Concentrated acids – Observe the following precautions when working with concentrated acids:

- Always wear appropriate PPE including lab coat, nitrile gloves, safety glasses and/or face shield.
- Never work with acids outside of a fume hood.
- Always add acid to water when preparing solutions.
- Identify all secondary containers appropriately with hazard labels.
- Neutralize acidic waste in a fume hood prior to disposal.

10.2 Reagents – Review the Material Safety Data Sheets (MSDS) for all reagents used in this procedure.

10.3 Digestion by-products – Mercury compounds are extremely hazardous and the acidification of samples containing reactive materials may result in the release of toxic gases. Always perform digestions in a fume hood.

11.0 Tables

Table 1- Stock Standards (all concentration in µg/mL)

Name				**Vendor**				**Catalog Number**		
ALS Custom Calibration Mix				Inorganic Ventures				ALSCHEMEX-CAL-13		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Aluminum	10		Calcium	1000		Magnesium	1000		Silver	10
Antimony	10		Chromium	10		Manganese	10		Sodium	1000
Arsenic	10		Cobalt	10		Molybdenum	10		Thallium	10
Barium	10		Copper	10		Nickel	10		Titanium	10
Beryllium	10		Iron	1000		Potassium	1000		Vanadium	10
Cadmium	1000		Lead	10		Selenium	10		Zinc	10
Aluminum				Inorganic Ventures				CGAL1		
Element	**Conc.**		**Element**							
Aluminum	10									
Strontium				Inorganic Ventures				CGSR1		
Element	**Conc.**		**Element**							
Strontium	1000									
Tin				Inorganic Ventures				CGSN1		
Element	**Conc.**		**Element**							
Tin	1000									
Instrument Check Standard 1 (2nd source)				SPEX				CL-ICS-1		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Aluminum	10		Cadmium	1000		Manganese	10		Vanadium	10
Antimony	10		Chromium	10		Nickel	10		Zinc	10
Arsenic	10		Cobalt	10		Selenium	10			
Barium	1000		Copper	10		Silver	10			
Beryllium	10		Lead	10		Thallium	10			
Calibration Standard 3 (2nd source)				SPEX				CL-CAL-3		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**			
Calcium	1000		Magnesium	1000		Sodium 1000				
Iron	1000		Potassium	1000						
Instrument Check Standard 5 (2nd source)				SPEX				CL-ICS-5		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Molybdenum	10		Strontium	10		Tin	10		Titanium	10
Aluminum (2nd source)				SPEX				CLAL2-2M		
Element	**Conc.**									
Aluminum	10									

Table 1- Stock Standards (cont.)

Name	**Vendor**	**Catalog Number**
Interference Check Solution	Inorganic Ventures	6020ICS-0A

Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Aluminum	1000		Chloride	10K		Molybdenum	20		Sodium	1000
Calcium	1000		Iron	1000		Phosphorus	1000		Sulfur	1000
Carbon	1000		Magnesium	1000		Potassium	1000		Titanium	20
Tuning Solution				SPEX				CL-TUNE-1		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Barium	10		Cobalt	10		Lithium 7	10		Thallium	10
Beryllium	10		Indium	10		Magnesium	10		Uranium	10
Cerium	10		Lead	10		Rhodium	10		Yttrium	10
Internal Standard Solution				VHG Labs				VHGLIS6020-500		
Element	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**		**Element**	**Conc.**
Bismuth	10		Indium	10		Scandium	10		Yttrium	10
Holmium	10		Lithium 6	10		Terbium	10			
Germanium (ISTD)				SPEX				PLGE9-2X		
Element	**Conc.**									
Germanium	1000									

Table 2 – Working Standards

Name	Stock Components	Amount added (µL)	Final Volume (mL)	Final Conc. (µg/L)
200/20,000 ppb Calibration Standard	ALS Custom Calibration Mix	1000	50	200/20K
	Aluminum	900		
	Strontium	1000		
	Tin	1000		
100/10,000 ppb Calibration Standard	ALS Custom Calibration Mix	500	50	100/10K
	Aluminum	450		
	Strontium	500		
	Tin	500		
10/1000 ppb Calibration Standard	ALS Custom Calibration Mix	50	50	10/1000
	Aluminum	45		
	Strontium	50		
	Tin	50		
5/500 ppb Calibration Standard	ALS Custom Calibration Mix	25	50	5/500
	Aluminum	22.5		
	Strontium	25		
	Tin	25		
2/200 ppb Calibration Standard	ALS Custom Calibration Mix	10	50	2/200
	Aluminum	9		
	Strontium	10		
	Tin	10		
0.5/50 ppb Calibration Standard	100 ppb Calibration Standard	250	50	0.5/50
0.2/20 ppb Calibration Standard	100 ppb Calibration Standard	100	50	0.2/20
Initial Calibration Verification (ICV)	Instrument Check Standard 1 (2nd source)	500	50	100/10K
	Calibration Standard 3 (2nd source)	500		
	Instrument Check Standard 5 (2nd source)	500		50
	Aluminum (2nd source)	450		
Continuing Calibration Verification (CCV)	ALS Custom Calibration Mix	500	50	100/10K
	Aluminum	450		
	Strontium	500		
	Tin	500		

Table 2 – Working Standards (cont.)

Name	Stock Components	Amount added (µL)	Final Volume (mL)	Final Conc. (µg/L)
0.2/20 ppb Low-Level CCV (LLCCV1)	100 ppb Calibration Standard	100	50	0.2/20
0.5/50 ppb Low-Level CCV (LLCCV2)	100 ppb Calibration Standard	250	50	0.5/50
2/200 ppb Low-Level CCV (LLCCV3)	100 ppb Calibration Standard	1000	50	2/200
Interference Check Solution A (ICSA)	Interference Check Solution	5000	50	2K/100K
Interference Check Solution AB (ICSAB)	Interference Check Solution	5000	50	100-110K
	ALS Custom Calibration Mix	500		
	Aluminum	450		
	Strontium	500		
	Tin	500		
Tune Check Standard	Tuning Solution	50	50	10
ISTD solution	Internal Standard Solution	5000	50	10
	Germanium (ISTD)	500		100
	Strontium	10		
ICPMS Spiking Solution	ALS Custom Calibration Mix	undiluted		

All standards are prepared in 1% HNO_3/0.5% HCl in class A volumetric flasks.

Table 3 - Instrument Acquisition Parameters			
Acquisition Mode	Spectrum		
Peak Pattern	1 point		
Replicates	3		
Sweeps/Replicate	50		
Mass	**Element Name**	**Tune Mode**	**Integration Time (sec)**
6	Li (ISTD)	No Gas	0.1000
9	Be	No Gas	0.1000
23	Na	He	0.1000
24	Mg	He	0.1000
27	Al	He	0.1000
39	K	He	0.1000
44	Ca	He	0.1000
47	Ti	He	0.1000
51	V	He	0.3000
52	Cr	He	0.3000
53	[V]	He	0.1000
55	Mn	He	0.3000
56	Fe	He	0.1000
57	Fe	He	0.1000
59	Co	He	0.3000
60	Ni	He	0.3000
62	Ni	He	0.3000
63	Cu	He	0.3000
65	Cu	He	0.3000
66	Zn	He	0.3000
68	Zn	He	0.3000
72	Ge (ISTD)	He	0.1000
75	As	He	1.0000
77	[As]	He	1.0000
78	Se	He	3.0000
88	Sr	He	0.3000
95	Mo	He	0.3000
97	Mo	He	0.3000
107	Ag	He	0.3000
108	[Cd]	He	0.1000
109	Ag	He	0.3000
111	Cd	He	1.0000
114	Cd	He	1.0000
115	In (ISTD)	He	0.1000
118	Sn	He	0.3000
119	Sn	He	0.3000
121	Sb	He	0.3000
123	Sb	He	0.3000
135	Ba	He	0.3000
137	Ba	He	0.3000
203	Tl	He	0.3000
205	Tl	He	0.3000

206	Pb	He	0.3000
27	Pb	He	0.3000
208	Pb	He	0.3000
209	Bi (ISTD)	He	0.3000

Table 4 – Interference Equations

Mass	**Equation**
6	(6)*1 – (7)*0.0813
51	(51)*1 + (52)*0.3524 – (53)*3.1081
75	(75)*1 – (77)*3.1278 + (78)*2.0177
78	(78)*1 – (76)*0.1869
114	(114)*1 – (108)*1.6285 + (118)*0.0149
115	(115)*1 – (118)*0.0149
208	(208)*1 + (206)*1 + (207)*1

Table 5 – QC Summary

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
MS tuning sample.	Prior to initial calibration	see section 7.3 of this SOP	Maintenance and/or retune instrument then reanalyze tuning solution
Initial Calibration (minimum 3 standards and a blank).	Daily initial calibration prior to sample analysis	r ≥ 0.995	Drop high point(s). Prepare new standards and/or recalibrate
Initial Calibration Verification (ICV)	After initial calibration and subsequent calibrations.	All analytes of interest within ±10% of expected value	Prepare new standards and/or recalibrate
Calibration Blank (ICB or CCB)	Beginning and end of sample run, after every 10 samples	All analytes of interest < ½ the reporting limit	Determine the cause and reanalyze samples with potential false positives
Interference Check Solutions (ICS-A and ICS-AB)	At the beginning of each daily analytical run and every 12 hours thereafter	ICS-A: All non-spiked trace analytes < ½ RL and others ±20% of true value ICS-AB: trace analytes within ±20% of true value	Reanalyze ICS; If still failing, determine cause, correct problem, recalibrate and reanalyze affected samples
Continuing Calibration Verification (CCV)	Beginning and end of sample run, after every 10 samples	All analytes of interest within ±10% of expected value	Correct problem then recalibrate and reanalyze all affected samples
Method Blank (MB)	One per preparation batch	All analytes of interest less than the greater of 2.2 x MDL or 10% of any reportable sample result	Determine the cause. Redigest and reanalyze samples with potential false positives
Blank Spike/Blank Spike Duplicate (BS/BSD)	One pair per preparation batch	Recoveries of 85% to 115%. RPD ≤ 20%	Determine the cause. Redigest and reanalyze samples with potential high or low bias
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One pair per 10 water samples One pair per 20 soil samples	Recoveries of 75% to 125%. RPD ≤ 20%	Determine the cause. Check results of Post-digestion spike and Dilution test. Redigest and/or reanalyze if appropriate
Post-digestion spike (PDS)	One per MS/MSD	Recoveries of 80% to 120%.	Determine the cause. Redigest and/or reanalyze if appropriate
Dilution test	One per MS/MSD	5x dilution should agree within ±10% of the original for analytes present at a concentration 25 times RL	Redilute and reanalyze both the dilution and the original sample.
Internal Standards (ISTD)	Every run	Samples, batch QC: IS counts 70-125% of reference value. CCBs and CCVs: IS counts 80-120% of reference value	Determine the cause. If due to matrix, dilute as necessary If not, recalibrate and reanalyze affected samples

Table 5 – QC Summary

QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**
Method Detection Limit (MDL) study	Annually	PQLs (= 3 x MDLs) that meet work order requirements	Instrument maintenance and/or retuning if PQLs are inadequate
Linear Range Determination (LDR)	Quarterly or whenever instrument response changes significantly	Upper Quantitation Limit (UQL) is determined from the highest standard for which the result is within 10% of the nominal value	Dilute the sample if any analyte of interest is above the UQL determined by the most recent LDR

840.2 Hg in Soil & Water

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840.2 HG IN SOIL & WATER

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Laboratory Director – Rick Bagan

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Analysis of Mercury in Soil and Water

1.0 Purpose

1.1 To outline the procedure used for determination of Mercury by Cold Vapor Atomic Absorption spectrometry.

2.0 References

2.1 ALSEV Quality Assurance Manual (QAM).

2.2 EPA Method 7470A (SW-846): Mercury in Liquid Wastes (Manual Cold Vapor Technique), revision 1, September 1994.

2.3 EPA Method 7471B (SW-846): Mercury in Solid or Semisolid Wastes (Manual Cold Vapor Technique), revision 2, January 1998.

2.4 EPA Method 245.1: Determination of Mercury in Water by Cold Vapor Atomic Absorption spectrometry, revision 3, 1994.

2.5 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

3.0 Definitions

3.1 Prep Batch – The basic unit for quality control. A prep batch consists of a group of up to 20 samples of the same matrix which are all digested on the same day. Each batch includes a Method Blank, Blank Spike, and Blank Spike Duplicate. In addition, a Matrix Spike and Matrix Spike Duplicate sample are included at a frequency of every 10 water samples and every 20 soil samples. The calibration levels are prepared along with the samples, using the same reagents. They do not undergo heating, however. A single calibration curve may be used for all batches of either matrix digested on the same day.

3.2 Method Blank – A quality control sample prepared by using DI water in place of the sample and taking it through entire digestion and analysis process. It is used to monitor contamination in the prep batch.

3.3 Blank Spike & Blank Spike Duplicate – Method Blanks with added Mercury. They are used to assess to accuracy and precision of the method.

3.4 Matrix Spike & Matrix Spike Duplicate – A Sample and Duplicate with added Mercury. It is used to monitor method accuracy and the effect of the sample matrix on Mercury recovery.

3.5 Method Detection Limit – A number (with units of concentration) generated according to the procedure described in 40 CFR, Part 136, Appendix B. The MDL is laboratory specific and must be determined annually. Theoretically, the MDL is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.6 Reporting Limit – The smallest amount of analyte that can be reliably quantified. For this method the reporting limit is the MDL times 3.

4.0 Apparatus and Materials

4.1 Teledyne CETAC Quick Trace M-7600 Mercury Analyzer with ASX-560 autosampler

4.2 Teledyne CETAC consumables kit for M-7600

4.2 17 x 100 mm polypropylene culture tubes

4.3 50 mL polypropylene digestion cups, Environmental Express

4.4 FilterMate filtration assemblies, Environmental Express

4.4 300 ml BOD bottles with stoppers

4.5 Volumetric flasks for standard and reagent preparation

4.6 Variable volume pipettors with ranges of 10 to 100uL, 100 to 1000uL, 1 to 5 mL, and 1 to 10 mL

4.7 Analytical balance accurate to 0.01 g, calibrated annually against ASTM Type 1 reference weights

4.8 Electric water bath capable of holding a temperature of 95 C

4.9 NIST traceable thermometer accurately displaying temperature up to 100 C

4.10 UHP Argon carrier gas.

5.0 Reagents and Standards

5.1 Stock Reagents and Standards

5.1.1 ASTM Type II water (ASTM D1193) drawn from ELGA Purelab Flex water system

5.1.2 Concentrated nitric acid, JT Baker instra-analyzed

5.1.3 Concentrated hydrochloric acid, JT Baker instra-analyzed

5.1.4 Concentrated sulfuric acid, JT Baker instra-analyzed

5.1.5 Stannous Chloride, JT Baker analyzed ACS

5.1.6 Potassium Persulfate, JT Baker analyzed ACS

5.1.7 Potassium Permanganate, JT Baker analyzed ACS

5.1.8 Hydroxylamine hydrochloride, JT Baker analyzed ACS

5.1.9 Sodium Chloride, BDH

5.1.9 Mercury standard, 1000 ug/mL, Inorganic Ventures

5.1.10 Mercury standard (2nd source), 1000 ug/mL, Ultra Scientific

5.2 Working Reagents and Standards

5.2.1 Sulfuric acid (0.5 N) – Slowly add 35 mL concentrated H_2SO_4 to about 2 L DI water in an acid dispenser bottle and bring to a final volume of 2.5 L. Mix well.

5.2.2 Stannous Chloride solution – Add 70 mL HCl to DI water in a one-liter volumetric flask. Add 100.0 g $SnCl_2$ and bring to volume. Add stir bar and place on stir plate until fully dissolved.

5.2.3 Potassium Persulfate solution – Add about 700 mL hot DI water to 45 g $K_2S_2O_8$ in a 1 L amber dispenser bottle, shake briefly and bring to a final volume of 900 mL. Place on stir plate until fully dissolved.

5.2.4 Potassium Permanganate solution – Add about 2.5 L DI water to 150 g KMnO4 in a 4 L amber dispenser bottle, shake briefly and bring to a final volume of 3 L. Shake vigorously to dissolve.

5.2.5 Sodium Chloride/Hydroxylamine solution – Add about 700 mL DI water to 102 g NaCl and 102 g $NH_2OH \cdot HCl$ in a 1 L amber dispenser bottle, shake briefly and bring to a final volume of 850 mL. Place on stir plate until fully dissolved.

5.2.6 Mercury calibration standard – Add 50 uL Mercury stock standard to DI water in a 50 mL volumetric flask. Bring to volume.

5.2.7 Mercury check standard – Add 50 uL Mercury stock standard to DI water in a 50 mL volumetric flask. Bring to volume.

6.0 Sample Collection and Preservation

6.1 Soil samples for metals analysis are collected in clean 4 ounce or larger jars.

6.2 Water samples for metals analysis are collected in clean 500 mL HDPE bottles and immediately preserved with HNO3.

6.3 Water samples for dissolved metals analysis are filtered as soon as possible upon collection using 0.45 um filters. Filtered samples are then preserved with HNO_3 in 500 mL HDPE bottles.

6.4 Samples are maintained @ 4 C, and should be analyzed within 28 days of collection.

7.0 Procedure

7.1 Sample Handling

7.1.1 Soil: Thoroughly mix sample in order to collect a representative sub sample. Pour off any supernatant liquid before mixing and discard any foreign objects (rocks, leaves, twigs).To determine dry sample weight, record weight of an empty pan, tare the pan and weigh 10 - 20 grams of the sample. Record the weight, and place pan in drying oven for at least 1 hour at approximately 100C. Weigh the pan with dry sample, and determine the percent dry weight as follows:

$$\% \text{ dry sample} = (C-B)/ A \times 100$$

where: A=wet sample
B=weight of pan
C=weight of pan + dry sample

7.1.2 Water: Shake sample well and pour out an appropriate amount into a clean BOD bottle. Use pH strips to verify that the sample has been properly preserved, and note as such in digestion logbook.

7.2 Calibration Standard/Sample Preparation

7.2.1 Calibration standards – Add 10, 25, 50, 100, 250, 500 and 1000 uL of the 1.0 ppm mercury calibration standard to a series of BOD bottles (include one unspiked bottle to serve as a calibration blank.) Dilute the standards to 100 mL with DI water. Add 5 mL 0.5N H_2SO_4 solution and 1.25 mL conc. HNO_3 to each bottle, followed by 15 mL $KMnO_4$ solution and 8 mL $K_2S_2O_8$ solution. (Do not heat in water bath)

7.2.2 Water samples – Measure 100 mL sample in a graduated cylinder and pour into a BOD bottle. Add the reagents as with the calibration standards, but wait 15 minutes after adding the $KMnO_4$ solution to see that purple color persists. If the color disappears, add 15 mL aliquots $KMnO_4$ solution until the color does persist for 15 minutes. (**Note**: If an additional 45 mL does not suffice, re-prepare the sample using a smaller sample amount. Also prepare an additional method blank (sec. 7.2.4.5) using the same amount of $KMnO_4$.) Stopper bottles and place in water bath to heat for 2 hours at 95C. Record the bath temperature and the start/stop times in the digestion logbook.

7.2.3 Soil samples – Weigh approximately 1 g (dry wt.) sample into a tared BOD bottle. Add 8 mL DI water plus 5 mL conc. HCl and 1.25 mL conc. HNO_3 solution to each bottle. Stopper and place bottles in 95C water bath for 2 minutes. Cool to room temperature before adding 50 mL DI water and 15 mL $KMnO_4$ solution. As with water samples, add enough $KMnO_4$ so that the color persists for 15 minutes. (See Note from sec. 7.2.2) Stopper bottles and place in water bath to heat for at least 30 minutes at 95C. Record the bath temperature and the start/stop times in the digestion logbook. Allow samples to cool to room temperature before adding another 50 mL DI water.

7.2.4 Quality control samples – For each preparation batch include the following QC samples (Note:

7.2.4.1 Quality Control Sample (QCS) – prepare along with the calibration standards using 250 uL of the 2nd source 1.0 ppm mercury check standard. (Do <u>not</u> heat in water bath)

7.2.4.2 Low Limit of Quantitation Check (LLQC) – prepare along with the calibration standards using 10 uL of the 1.0 ppm mercury calibration standard. (Do <u>not</u> heat in water bath)

7.2.4.3 Initial and Continuing Calibration Verification (ICV,CCV) – prepare along with the calibration standards using 250 uL of the 1.0 ppm mercury calibration standard. (Do <u>not</u> heat in water bath)

7.2.4.4 Initial and Continuing Calibration Blank (ICB,CCB) – prepare in the same manner as the ICV, but do not add mercury standard. (Do <u>not</u> heat in water bath)

7.2.4.5 Method Blank (MB) – For each matrix prepare a blank that is carried through all the steps specific to the matrix (soil or water).

7.2.4.6 Blank Spike/Blank Spike Duplicate (BS/BSD) – For each matrix prepare a pair of spiked blanks using 250 uL of the 1.0 ppm mercury check standard, to be carried through all the steps specific to that matrix (soil or water).

7.2.4.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – For each matrix (at frequency noted in sec. 3.1) prepare a pair of spiked samples using 250 uL of the 1.0 ppm mercury check standard and sample amounts equal to the original sample, to be carried through all the steps specific to that matrix (soil or water).

7.2.4.8 Post Digestion Spike (PDS) – For each matrix, spike an aliquot of the digestate from the sample used for the MS/MSD with an amount of the mercury check standard that will result in the equivalent spike concentration as the MS/MSD.

7.2.4.9 Dilution Test (DT) – For each matrix, run a 1:5 dilution of the sample used for the MS/MSD.

7.2.5 When all the samples have cooled to room temperature, add 6 mL NaCl/ $NH_2OH \cdot HCl$ solution to each bottle (including the calibration and verification standards.)

7.2.6 Pour sample digests into polypropylene culture tubes for analysis, making dilutions as needed. Samples with significant suspended solids should be filtered first. Pour the sample into a 50 mL digestion cup and push a FilterMate assembly to the bottom of the tube.

7.3 Instrument Operation & Data Collection

7.3.1 Open an instrument session by double clicking the QuickTrace icon. This will automatically turn on lamp and begin a warmup period of about 30 minutes. If necessary, empty the waste container and refill the 2 L rinse container with 1% HNO3 / 1% HCl.

7.3.2 Begin argon flow by opening the valve on the gas regulator panel. Secure the peristaltic pump tubing in place and clamp down the pressure shoes. Check to see that the Hg vapor tube is disconnected from the gas-liquid separator (GLS).

7.3.3 Click on the Instrument Control button. Select the Autosampler tab to turn on the pump. Select the Analyzer tab to set the gas flow to 100 mL/min and turn on the pump. Place the reagent capillary tube in a flask containing 2% HNO_3 / 2% HCl rinse solution. Move the autosampler probe to a beaker containing the same solution.

7.3.4 While the instrument is warming up, select New From in the File drop-down to create a new worksheet using a previous worksheet as a template. The template should include the calibration followed by QCS, LLQC, and ICV/ICB. Edit the sequence table to include the current batch samples with required QC (including post-digestion spike and dilution test), and a CCV/CCB pair for every 10 runs and at the end of the sequence. Include all standard IDs.

7.3.5 When the warmup period is complete, the GLS button will become available. Click on the icon in order to ramp up gas flow & pump speed to facilitate wetting the GLS post. Temporarily release a pressure shoe on the drain tubing in order to allow bubbles to rise in the GLS, thereby wetting the post.

7.3.6 Reclamp drain tubing pressure shoe to clear excess liquid from the GLS. Attach the Hg vapor tube to the GLS and place the reagent tube in the bottle containing $SnCl_2$ solution.

7.3.7 Open the Method Editor and select the Analyze a Sample button to check the peak profile of a midpoint calibration standard. If the peak looks normal and integration times appear to be correct, close the editor and click GO to begin the sequence. Once the calibration is complete inspect the curve to be sure it meets the minimum acceptable correlation coefficient of 0.997 before proceeding. The highest calibration level (10 ppb) may be dropped if necessary. (Right-click on the calibration curve to inspect the data and reject the 10 ppb level.)

7.3.8 Upon completion of the sequence, click on Window drop-down menu and select View Results to scan through the peak profiles of all the samples. Rerun any samples with severely distorted peak shapes, along with dilutions for any samples above the calibration range. When all data is satisfactory, save the worksheet and print a copy of the data.

7.3.9 Return the reagent tube to the rinse solution flask and move the autosampler probe to the rinse solution beaker. Rinse for about 5 minutes then remove the reagent tube from solution and move the probe to the Park/Up position. When all solution has cleared from the system, close the instrument session, release the pump tubing & Hg vapor tube, and close the Argon valve.

8.0 Data Analysis & QC Criteria

8.1 Calculations

8.1.1 Final mercury concentration is calculated as follows:

Soil: IC x (V/W) x DF = mg/kg Hg

Water: IC x DF = ug/L Hg

where IC = instrument concentration (ug/L),

V = nominal digest volume (0.1 L),

W = amount of soil (dry wt.) in grams,

DF = dilution factor

8.1.2 Percent recovery and Relative percent difference are calculated as follows:

Blank Spike: % Recovery = (Spike result/Expected spike result) x 100

$$\text{Matrix Spike: \% Recovery} = \frac{(\text{Spike result} - \text{Sample result}) \times 100}{\text{Expected spike result}}$$

$$\text{RPD} = \frac{(\text{Spike Recovery} - \text{Spike Duplicate Recovery}) \times 100}{(\text{Spike Recovery} + \text{Spike Duplicate Recovery})/2}$$

8.2 Batch QC Acceptance Criteria

8.2.1 The QCS should fall in the range of 90 to 110% of the expected value.

8.2.2 The LLQC should fall in the range of 70 to 130% of the expected value.

8.2.2 The ICV should fall in the range of 95 to 105% of the expected value and subsequent CCVs should fall in the range of 90 to 110%. The ICB and subsequent CCBs should be less than the MDL.

8.2.3 Any samples with mercury levels above the highest point of the calibration should be rerun at a dilution that will give a result in the upper half of the calibration range.

8.2.4 Method Blanks should be less than 2.2 times the MDL or less than 1/10th of the concentration of any sample in the batch.

8.2.5 Blank Spikes should fall in the range of 85 to 115% and RPDs should be 15% or less.

8.2.6 Matrix Spikes should fall in the range of 75 to 125% and RPDs should be 20% or less.

8.2.7 If the Matrix Spike recovery is outside of the acceptance range, check the results of the post-digestion spike and dilution test to confirm matrix effects. The post-digestion spike should be recovered to within 80 to 120% of the known value, and the 1:5 dilution should agree within 10% of the original determination.

8.3 Method Detection Limits should be determined on an annual basis at a minimum. Follow the procedure outlined in section 9.4.2 of EPA method 245.1

8.4 Performance Evaluation Samples are analyzed on an annual basis for each matrix.

9.0 Records Management

9.1 Standards Log – Stock standards are assigned a T-code ID and logged into the lab-wide Standard Solutions Log. A copy of the manufacturer certificate of analysis is placed in the lab COA binder. Standard containers are labelled with the ID and dates of receipt and opening.

9.2 Metals Standards Log – Working standards are assigned an S-code ID and logged into the Metals Standards Preparation Log. Standard containers are labelled with the ID and expiration date.

9.3 Metals Reagent Log – Stock reagents and reagent solutions prepared from stock are assigned an R-code ID and logged into the Metals Reagent Log. Stock reagent containers are labelled with the ID and dates of receipt and opening. Reagent solutions are labelled with the ID and expiration date.

9.4 Digestion Log – Information pertaining to each digestion batch is recorded in a bound notebook. It should include a list of the samples included in the batch along with standard and reagent codes, water bath temperature, digestion start/stop times, and analyst initials & date.

9.5 Data – Instrument raw data is saved on the instrument computer and a hardcopy is used to create Excel cover sheets summarizing sample final concentrations, spike recoveries, etc. The data is reviewed by the analyst and copies of the cover sheets are initialed, dated and placed in project folders for secondary review. The data is then uploaded to the LIMS. The hardcopy data and copies of the cover sheets are placed in a file folder and kept in the instrument data archive.

10.0 Safety

10.1 Concentrated acids – Observe the following precautions when working with concentrated acids:

- Always wear appropriate PPE including lab coat, nitrile gloves, safety glasses and/or face shield.
- Never work with acids outside of a fume hood.
- Always add acid to water when preparing solutions.
- Identify all secondary containers appropriately with hazard labels.
- Neutralize acidic waste in a fume hood prior to disposal.

10.2 Reagents – Review the Material Safety Data Sheets (MSDS) for all reagents used in this procedure.

10.3 Digestion by-products – Mercury compounds are extremely hazardous and the acidification of samples containing reactive materials may result in the release of toxic gases. Always perform digestions in a fume hood.

720.0 Inorganic Ions in Water by IC

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720.0 INORGANIC IONS IN WATER BY IC

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Approved By: _________________Rick Bagan________ Date: ____8/13/2018___

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Determination of Inorganic Ions in Water by Ion Chromatography

1.0 Purpose

1.1 To outline the procedure for the determination of the concentration of the following inorganic anions by ion chromatography: Bromide, Chloride, Fluoride, Nitrate, Nitrite, ortho-Phosphate, Sulfate.

2.0 References

2.1 ALSEV Quality Assurance Manual (QAM).

2.2 US.EPA Method 300.0 A Revision 1.0 US-EPA Environmental Monitoring Systems, Cincinnati, OH. 45268

2.3 Standard Methods for the Examination of Water and Wastewater, Method 4110B, “Anions by Ion Chromatography”, 20th Edition of Standard Methods (1998)

2.4 40 CFR, Part 136, Appendix B

3.0 Definitions

3.1 Analytical Batch – The basic unit for quality control. An analytical batch represents samples, which are analyzed together with the same method, same lots of reagents and same steps in common to each sample, with the same time period or within one week. The maximum batch size is 20 samples.

3.2 Initial Calibration Curve (ICal) – A minimum of 3 different standard concentrations, and a blank which bracket the anticipated concentration range, made from a stock solution. The curve must have a correlation coefficient of 0.995 or higher

.

3.3 Initial Calibration Verification (ICV) – The first mid-range working standard used to verify that the instrument is functioning correctly and that the initial calibration is still valid.

3.4 Continuing Calibration Verification (CCV) – Any subsequent mid-range working standards diluted from the stock standard used to verify that the analytical system is operating in a manner comparable to that at the time of initial calibration.

3.5 Initial Calibration Blank (ICB)– A blank used to verify the calibration curve, that is run immediately before the ICV and must have a value that is less than the detection limit. If the ICB fails, then it should be re-analyzed. If the re-analysis fails, the instrument must be recalibrated.

3.6 Continuing Calibration Blank (CCB) – A calibration check blank used to verify the calibration curve that is run after every 10 samples and at the end of every sample run. The CCB must have a value that is less than the detection limit. If the CCB fails, all samples up to a preceding acceptable ICB or CCB must be rerun.

3.7 Second Source Standard Solution – A calibration check standard prepared from a source independent of the primary calibration standard. It is used to verify the accuracy of the initial calibration curve.

3.8 Method Blank (MB)– An artificial sample designed to monitor the introduction of artifacts into the analytical scheme. The method blank is taken through each step of the analysis.

3.9 Blank Spike (BS) – A quality control sample prepared by adding a second source standard solution to a blank matrix and carried through the entire analysis process

3.10 Matrix Spike (MS) – A quality control sample prepared by adding a second source standard solution to a sample matrix and carried through the entire analysis process.

3.11 Sample Duplicate (DUP) – A replicate of a sample used to determine the precision of the analytical method for the sample matrix.

3.12 Reporting Limit – The smallest amount of analyte that can be detected and reliably quantified and is based on the MDL.

3.13 Method Detection Limit (MDL) – A number, with units of concentration, generated according to the procedure described in 40 CFR, Part 136, Appendix B. The MDL is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

4.0 Apparatus and Materials

4.1 Analytical Instruments

4.1.1 Ion Chromatograph- Dionex Series Dx-100. Complete analytical system including Anion analytical column(Dionex AS9-SC), guard column, suppressor device, conductivity detector, and Dionex PeakNet 5.2 Data Chromatography Software.

4.2 Sample Preparation Equipment

4.2.1 Balances

4.2.1.1 Analytical Balance, capable of weighing to 0.1 mg.

4.2.1.2 Top loading balance, capable of weighing to 0.01 g.

4.2.2 100mL Volumetric flasks, with caps.

4.2.3 50 mL digestion tubes

4.2.4 5mL Plastic sample vials with filter caps.

4.2.5 VWR Variable Volume Pipettors with ranges of 0.01mL – 10.0mL.

4.2.6 VWR Macro tips for variable volume pipettors, free of trace metals.

4.2.7 1000 mL Volumetric flask, with cap.

4.2.8 Membrane filter paper pore size 0.45 µm.

4.2.9 Flip filters

4.2.10 FilterMate filtration devices

4.2.11 Miscellaneous glassware typically used in an analytical laboratory such as funnels, spatulas, weighing pans, beakers, volumetric flasks, desiccators, magnetic stirrers, and stir bars.

5.0 Reagents

5.1 Deionized water (DI) – Drawn from PureLab Flex water system.

5.2 Eluent solution- Purchased as a concentrate from vendors, and diluted approximately 10mL into 1L prior to adding to eluent container.

5.3 Stock standard solutions- Stock standard solutions purchased as certified solutions.

5.4 10M NaOH solution for adjusting pH: Weigh 100g NaOH into beaker. Transfer to a 500mL bottle and add 250mL DI water and mix.

6.0 Sample Collection, Preservation and Handling

6.1 Samples are normally collected in glass or plastic containers with Teflon lines closures.

6.2 Samples are shipped in coolers with coolant and appropriate packaging to prevent cross contamination and breakage.

6.3 Sample preservation and holding times are presented in Table 1. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment.

6.4 Samples and the extracts must be stored at 4° C until analyzed.

7.0 Procedure

7.1 Calibration

7.1.1 For operation, calibration or general use and care of the ion chromatograph, reference Dionex Dx-100 Operator’s Manual. Standard operating conditions are indicated in Table 2.

7.1.2 Prepare at least 3 calibration standards in plastic containers. The lowest standard should be equivalent to the reporting limit or be near, yet above the MDL. The standards must bracket the anticipated sample concentration range. The analyte concentrations for general use are prepared from the multi-element concentrate as follows.

Standard 1	0.2 mg/L
Standard 2	0.5 mg/L
Standard 3	1 mg/L
Standard 4	2 mg/L
Standard 5	5 mg/L
Standard 6	10 mg/L
Standard 7	15 mg/L
Standard 8	20 mg/L

7.1.3 If the correlation coefficient is >0.995, the calibration is assumed to be linear.

7.1.4 The calibration curve is verified using a mid-level continuing calibration standard. This standard is analyzed at the beginning and at the end of the analytical sequence and also after every 10 samples within the analytical sequence. If initial calibration check standards are within +/- 10% of the expected values, sample analysis can proceed.

7.1.5 Calibration standards and all QC samples are to receive the same preparation as samples once the standards have been made to the appropriate concentration.

7.2 Sample and Standard Preparation

7.2.1 The analytical batch consists of 20 samples. The following quality assurance samples must be analyzed with each batch:

1 **method blank** per day at the rate of 1 per batch or with each extraction event, whichever is more frequent.

1 **blank spike** per sample batch.

1 **blank spike duplicate** per sample batch.

1 **duplicate** sample per batch.

1 **matrix spike** sample per batch.

7.3 Analysis

7.3.1 Table 2 summarizes the recommended operating conditions for the ion chromatograph

7.3.2 Load and inject a fixed amount (5 mL) of well mixed standard or sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Samples that are out of range of the calibration must be diluted to within the calibration range and the dilution factor used to calculate the final concentration.

7.3.3 Using an instrument blank that received the same treatment as the samples, establish baseline stability.

7.3.4 Read all calibration standards, check standards and samples as described above, making sure they meet all QC acceptance criteria, as described previously and found in Appendix 1.

7.4 Calculations

7.4.1 Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. If a sample has been diluted, multiply the response by the appropriate dilution factor.

7.4.2 Report only those values that fall below the highest calibration standards.

7.4.3 Report values to 2 significant figures, but not more accurate than the least accurate unit of the low calibration standard.

8.0 Quality Control

8.1 On-going quality control

8.1.1 Quality control acceptance criteria is given in Appendix 1.

8.1.2 The method blank must show a non-detect for the analytes of interest. If the method blank fails to meet acceptance criteria, then diagnose the problem and take corrective action. Analyze the method blank sample for the analytical batch prior to the duplicates and field samples

8.1.3 Analyze a duplicate sample.

8.1.4 Calculate the relative percent difference (RPD) for duplicate analyses using the following equation, where D_1 and D_2 represent the results from duplicate analyses:

$$RPD = |D_1 - D_2| / (D_1 + D_2) / 2 \text{ X } 100$$

8.1.5 Compare the RPD with the current acceptance criteria for this procedure. If the RPD meets the acceptance criteria of 25%, all the samples in the analytical batch are acceptable. If the RPD fails to meet criteria, diagnose the problem and discuss with laboratory director or QC Officer to determine if the analytical batch is to be reported.

8.1.5 Analyze a blank and sample each spiked with a known amount of analyte then calculate the percent recovery of the spike. Recoveries should be +/- 10% of the expected value. If an analyte falls outside of the expected range, the source of the problem should be identified and resolved before continuing the analysis.

8.2 A method detection limit determination is performed using the procedure described in 40 CFR, Part 36 Appendix B.

8.2.1 The method detection limit determination is to be performed at least annually to demonstrate confidence levels. Project specific plans may require additional determinations at specified frequencies.

8.3 Nonconformance and Corrective Action

8.3.1 Any discrepancy affecting the quality of the data for any sample is documented on a nonconformance corrective action report (NCAR) or within the project file.

9.0 Records Management

9.1 Initial calibration curve data are maintained in the instrument ICAL files

9.2 Sample results and the QC results for each analytical batch are maintained in the instrument run files.

9.3 After an independent data review has been completed, a copy of the pertinent sample data is filed in the appropriate client project files.

10.0 SAFETY

This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1:	**Hazards**	**Preventative Measures**
Handling samples and reagents	Preserved samples may be acidic	Read appropriate Safety Data Sheets. Wear proper protective equipment including gloves, lab glasses, and lab coat.
Job Task #2:	**Hazards**	**Preventative Measures**
Handling glassware	Breakage possible	Handle with care. Replace broken or chipped glassware.
Job Task #3:	**Hazards**	**Preventative Measures**
Using gas cylinders and pressurized containers	Highly pressurized	Follow safety guidelines for handling gas cylinders. Secure properly. Move with a hand truck.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

TABLE 1 Sample Preservation and Holding Times

Analyte	Preservation	Holding Time
Bromide	None	28 days
Chloride	None	28 days
Fluoride	None	28 days
Nitrate	Cool to 4C	48 hours
Nitrite	Cool to 4C	48 hours
Ortho-Phosphate	Cool to 4C	48 hours
Sulfate	Cool to 4C	28 days

TABLE 2 Standard Operating Conditions

STANDARD CONDITIONS

Column: Dionex AS9-SC

Detector: Conductivity Cell

Pump Rate: 1 ml/min

Eluent: As specified in sec. 5.2

Sample Loop: 50 uL

APPENDIX 1

Ion Chromatographic analysis of Inorganic Anions
Acceptance Criteria for Quality Control

	% Recovery	Relative % Difference
Calibration Verification	90-110	
Blank Spike and Duplicate	90-110	25
Sample Duplicate		25
Matrix Spikes	80-120	

ALS Standard Operating Procedure

DOCUMENT TITLE:	TOTAL ORGANIC CARBON IN WATER
REFERENCED METHOD:	EPA 9060A; SM 5310C
SOP ID:	GEN-TOC
REVISION NUMBER:	14
EFFECTIVE DATE:	9/15/2015

ALS-Kelso SOP Annual Review Statement

SOP Code: GEN-TOC
Revision: 14

An annual review of the SOP listed was completed on (date): _____

☐ The SOP reflects current practices and requires no procedural changes.
Supervisor: hlj Date: 2/12/18

☒ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	Description of Revision Needed	Date Procedure Change Implemented	Supervisor Initials Indicating Approval of Revision
11.4.5.1	Only samples are run in dudplicate. QA samples such as matrix spikes and duplicate matrix spikes are only analyzed with a single analysis for each.	2/12/18	hlj
11.4.6.1	Only samples are run in quadruplicate. QA samples such as matrix spikes and duplicate matrix spikes are only analyzed with a single analysis for each.	2/12/18	hlj

ALS-Kelso SOP Annual Review Statement

SOP Code: GEN-TOC
Revision: 14

An annual review of the SOP listed was completed on (date): 3/9/17

☒ The SOP reflects current practices and requires no procedural changes.
Supervisor: HLJ Date: 3/9/17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	Description of Revision Needed	Date Procedure Change Implemented	Supervisor Initials Indicating Approval of Revision

TOTAL ORGANIC CARBON IN WATER

EPA 9060A; SM 5310C

ALS-KELSO

SOP ID:	GEN-TOC	Rev. Number:	14	Effective Date:	9/15/2015

Approved By: Harvey Jacky Date: 8/24/15
Department Supervisor/Technical Director – Harvey Jacky

Approved By: Lee Wolf Date: 8/24/15
QA Manager – Lee Wolf

Approved By: Date: 8/24/15
Laboratory Director – Jeff Grindstaff

Issue Date: ___________ Doc Control ID#: ____________ Issued To: ____________

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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TOTAL ORGANIC CARBON IN WATER

1. SCOPE AND APPLICATION

1.1 This procedure is applicable to the determination of Total Organic Carbon (TOC) in drinking, surface and saline waters, domestic and industrial wastewater using methods EPA 9060A, and Standard Methods 5310C, 20th Edition. The procedure may also be extended to certain domestic or industrial wastes.

1.2 This procedure may be modified for quantification of Dissolved Organic Carbon (DOC) where TOC is determined from a filtered sample.

1.3 Normal operating parameters (i.e. 1 ml sample loop) yield a Method Reporting Limit (MRL) of 0.5 mg/L C. A 5 ml sample loop may be used to lower the MRL to 0.1 mg/L C. The data quality objectives for target analytes in water are presented in Table 2 and in the ALS Kelso DQO Table.

1.4 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

1.5 Total Organic Carbon (TOC) is determined by measuring carbon dioxide released by chemical oxidation of the non-purgeable organic carbon in the sample. After the sample has been acidified and purged of inorganic carbon, sodium persulfate, a strong oxidizer, is added. This oxidant quickly reacts with non-purgeable organic carbon in the sample at 100°C to form carbon dioxide. When the reaction is complete, the carbon dioxide is purged from the solution, concentrated by trapping then thermally desorbed (200°C) and carried into a non-dispersive infrared detector that has been calibrated to directly display the mass of carbon dioxide detected. The resulting carbon mass in the form of carbon dioxide is the equivalent to the mass of organic carbon originally in the sample.

2.1. Total Inorganic Carbon is determined by carbon dioxide released by acidification of a sample. The pH of the sample is lowered; carbonate and bicarbonate ions are converted to dissolved carbon dioxide. This carbon dioxide is purged from the solution, concentrated by trapping, and detected as described for TOC.

3. DEFINITIONS

3.1. Laboratory Control Sample (LCS) - a solution of prepared in the laboratory which goes through all steps of the analysis that a sample does, and is used to determine if the analysis is in control.

3.2. Method Blank (MB) - a solution of the laboratory prepared deionized water that is carried through analysis like a sample, to serve as a measure of contamination associated with laboratory storage, preparation, or instrumentation.

3.3. Continuing calibration blank (CCB) - a blank solution of deionized water. CCB's are analyzed to verify that the instrument has not become contaminated during the course of the analytical run.

3.4. Continuing calibration verification standard (CCV) - a solution of prepared in the laboratory at approximately the midpoint of calibration curves. CCV's are analyzed to verify that the instrument performance has not changed during the course of the analytical run.

3.5. Independent Calibration Verification (ICV) - Initial calibration verification standards which are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

3.6. Sample Duplicate - a second aliquot of a sample that is treated exactly the same throughout laboratory analytical procedures. The purpose is to verify the precision associated with the laboratory procedures. The Relative Percent Difference (RPD) should not exceed 20%.

3.7. Matrix Spike - aliquots of sample to which known amounts of an analyte of interest has been added. These are treated exactly the same throughout laboratory analytical procedures. The purpose of a matrix spike is to determine whether the sample matrix contributes bias to the analytical results.

3.8. Analytical Run Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with the instrument calibration or calibration verification followed by samples interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Refer to the SOP for *Sample Batches* for description of applicable batching procedures.

4. INTERFERENCES

4.1. Carbonate and bicarbonate carbon are interferences under the terms of this test and must be removed or accounted for in the final calculations

4.2. This procedure is applicable only to homogenous samples that can be injected reproducibly by microliter type syringe or pipette. The opening of the syringe or pipette limits the size of particles which may be included in the samples (both the Model 700 and Model 1010 analyzers can analyze samples with suspended solids up to 500 microns diameter.

4.3. Positive bias may be caused by contaminants in the gas, dilution water, reagents, glassware, or other sample processing hardware. The use of high purity reagents and gases help minimize interference problems. Materials may be demonstrated to be free from interference by running reagent blanks

4.4. Interference by non-CO2 gases: The infrared detector is sensitized to carbon dioxide and accomplishes virtually complete rejection of response from other gases which absorb energy in the infrared region. Trapping and desorption of carbon dioxide on the molecular sieve trap isolates the component of interest and allows the complete absence of interference in the system from gases other than carbon dioxide.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS Kelso safety policies, approved methods and in MSDSs where available. Refer to the ALS Kelso Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Always wear chemical eye, skin, and clothes protection when handling samples or working with reagents.

5.4. Sodium Persulfate is a strong oxidizer and should be handled with extreme care.

5.5. Phosphoric Acid is a corrosive material should be handled with extreme care.

5.6. Potassium Biphthalate and Sodium Carbonate are chemical irritants and may cause eye burns.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. For most accurate analyses, sampling containers should be free of organic contaminants.

6.2. Sampling and storage of samples in glass bottles is preferable. If this is not feasible, sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

6.2.1. **Note:** A brief study performed at the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

6.3. For samples requiring very low-level TOC analysis (below about 500 ppb C) attention to limiting contamination may be required. If possible, rinse bottles with sample before filling and carry field blanks through sampling procedure to check for any contamination that may occur. Collect and store samples in glass bottles protected from sunlight and seal with TFE-backed septa. Use certified clean sample vials for sampling and analysis. However if certified clean containers are not available or are found to be cleaned insufficiently further cleaning may be required. If necessary before use, wash bottles with acid, seal with Aluminum foil, and bake at 400°C for at least one hour. Wash un-cleaned TFE septa with detergent, rinse repeatedly with organic free water, wrap in Aluminum foil and bake at 100°C for one hour. Check performance of new or cleaned septa by running appropriate blanks. Preferably use thick silicone rubber-backed TFE septa with open ring caps to produce a positive seal. Less rigorous cleaning may be acceptable if the concentration range is relatively high. Check bottle blanks to determine effectiveness or necessity of cleaning.

6.4. Because of the possibility of oxidation or bacterial decomposition of certain components in aqueous samples, the time between sample collection and analysis should be minimized. In addition, the samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

6.5. In situations where analysis cannot be performed within two hours (2 hours) of sampling, the sample must be acidified (pH < 2) with Phosphoric or Sulfuric acid. Once preserved, samples must be analyzed within 28 days. Note that acid preservation invalidates any inorganic carbon determination on the samples.

6.6. Samples requiring DOC analyses should be filtered through a prewashed 0.45 micron glass microfiber membrane filter prior to acid preservation. A DI water filter blank should also be included with the filtration batch to determine potential for sample contamination from filter or filtration apparatus.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent (laboratory deionized) water, ASTM Type II.

7.2. Potassium Biphthalate (KHP) stock solutions:

7.2.1. 1000 ppm C stock solution is prepared by adding 2.128 g of KHP (previously dried to a constant weight at 105°C) into a 1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 1.0 ug C per ul.

7.2.2. 5000 ppm C stock solution is prepared by adding 10.64 g of KHP (previously dried to a constant weight at 105°C) into a 1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 5.0 ug C per ul.

Note: Stock solution has a shelf life of six months after preparation. Sodium oxalate and acetic acid are not recommended as stock solutions.

7.2.3. Linear range verification solutions at a minimum 5 concentrations, typically 0.5-50 ppm are prepared by diluting appropriate amounts of the 1000 mg/L stock standard to 100 mls with reagent grade water. These standards should be prepared fresh each time a multi-point calibration is performed.

7.3. Sodium Carbonate Stock solution (1000 ppm C) - Prepare stock solution by adding 8.826 g of Na_2CO_3 (previously dried to a constant mass at 105°C) to a 1000 ml volumetric flask. Dilute to volume with reagent water. Solution contains 1.0 ug C per ul.

7.4. Sodium Persulfate (250 g/L) - Prepare solution of sodium persulfate by dissolving 250g $Na_2S_20_8$ into preheated reagent water (1 liter volume). Reagent has a shelf life of one month.

Note: Reagent water is heated until solution just comes to a boil. Once reagent water has come to a boil, remove from heat and add sodium persulfate (250 g). Stir until persulfate goes into solution, then immediately cool by running water over the outside of beaker. This procedure purifies the $Na_2S_20_8$ solution by reducing TOC content of reagent water. Once cool, place the Model 700 purge lines in solution to remove any CO2 from oxidation of organics. Alternatively, dissolve sodium persulfate (250g) in 1L reagent water and purge with nitrogen for 5-10 minutes before use.

7.5. Phosphoric Acid (5%) - Prepare 5% by volume solution of phosphoric acid by adding 59 ml of ACS reagent grade 85% H_3PO_4 to reagent water (1 liter total volume). Reagent has a shelf life of one month.

7.6. The ICV is prepared by diluting 0.8 mL of 5000 ppm KHP stock solution to 200 mL DI water in a class A volumetric flask. Resulting concentration is 20.0 ppm. For low level analysis, dilute 2.0 mL of the 1000 ppm KHP stock solution to 1L DI water in a class A volumetric flask. Resulting concentration is 2.0 ppm. The shelf life is 6 months.

7.7. Continuing Calibration Verification (CCV) - The CCV is prepared by diluting 5.0 mls of 5000 ppm KHP stock solution (see 8.2) to 1000 mls in a class A volumetric flask. Resulting concentration is 25.0 ppm. For low level analysis, dilute 5.0 mls of the 1000 ppm KHP stock solution to 1000 mls in a class A volumetric flask. The shelf life is 6 months.

7.8. Laboratory Control Sample (LCS) - The LCS is prepared from Demand APG (Analytical Products Group). The true value is determined based on the lot number of the standard. The resulting standard has a shelf life of six months unless APG has a predetermined expiration date which expires prior to six months.

7.9. Gas Service: Nitrogen.

8. APPARATUS AND EQUIPMENT

8.1. TOC analyzer: Teledyne -Tekmar, Model TOC Fusion, S/N: US10165001.

8.2. Model 1010 Total Organic Carbon Analyzer: Utilizes classic persulfate oxidation method. (O.I. Analytical)

8.3. Autosampling Capability, Model 1010: 88-sample capacity, (model 1051).

1.6 Whatman 0.45µm glass microfiber membrane filter, or equivalent.

8.4. Apparatus for blending or homogenizing samples.

8.5. Note: Homogenization: Prior to analysis, the sample is thoroughly mixed by shaking the sample in the bottle rather than blending the sample. The concern is for possible contamination from the blender. It is not considered that this will misrepresent the true best average of the sample. The Model 1051 autosampler has magnetic stirring capability that homogenizes the sample prior to injection.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. For the most reliable performance of the instrument, the following schedule of routine maintenance is suggested (or as needed):

9.2.1. Weekly:

9.2.1.1. Replace gas cylinder
9.2.1.2. Adjust IR “zero”
9.2.1.3. Leak-check the carrier and purge gases
9.2.1.4. Check tube end fitting connections

9.2.2. Quarterly:

9.2.2.1. Replace or clean the permeation tube
9.2.2.2. Clean the digestion vessel
9.2.2.3. Check indicating drying tube
9.2.2.4. Check sample pump

9.2.3. Semi-annually:

9.2.3.1. Clean NDIR cell

9.2.4. Annually:

9.2.4.1. NDIR linearization check

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the ALS-Kelso *SOP for Training Procedure (*ADM-TRAIN*)*, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Turn on the nitrogen gas flow and confirm delivery pressure (50-60psi). Maintain this delivery pressure. If pressure drops to below 15 psi, the instrument will automatically shut down.

11.2. Initial Power Up

11.2.1. Turn on power to the Model 1010 analyzer, Model 1051 autosampler and computer using the main power switches.

11.2.1.1. During the Model 1010 power-up, listen for a series of beeps to determine the status of the instrument. The beep sequence is 1 beep= system startup, 2 beeps= CMOS check passed and 3 beeps= Firmware ready. If the beeps are not heard, contact OI Analytical Service Department for assistance.

11.2.1.2. Log into the WinTOC program. The user name is “CAS” and the password is “CAS”. Select TOC1 for operation.

11.2.2. To obtain a stable baseline, a reagent blank sequence must be started.

11.2.2.1. From the ‘Setup’ drop down, select WinTOC output. Change file names to reflect the date of analysis.

11.2.2.2. Select the reagent blank sequence from the ‘Sequence’ drop down.

11.2.2.3. Ensure that the most recent calibration check is selected from the ‘Calibration’ drop down.

11.2.2.4. Ensure that the TOC method is selected from the Database menu.

11.2.2.5. Click the start button on the status screen to begin the reagent blank sequence.

11.2.2.6. A stable baseline is obtained when the area counts are in the range of 50 to 500, and the last three area counts are within 50 counts of each other.

11.2.2.7. Once these criteria are met, abort the reagent blank sequence by clicking ‘Abort’ on the status screen.

11.2.3. An analysis sequence may now be started.

11.2.3.1. Select the run sequence desired from the ‘Sequence’ drop down.

11.2.3.2. Enter samples and standards in the selected run sequence.

11.2.3.3. Load tray into autosampler and click start on the status screen.

11.3. Calibration

11.3.1. The infrared detector response has been linearized and is fixed. A single point calibration verification is performed. Consult page 63 of the model 1010 user manual, for the proper calibration procedure.

11.3.1.1.For routine analyses (i.e. 1 ml sample loop) a 25 ppm standard is used for calibration.

11.3.1.2.For low level analyses (i.e. 5 ml sample loop) a 5 ppm standard is used for calibration.

11.3.2. Although the infrared detector response has been linearized, a series of five linear range verification standards are analyzed annually to confirm that the instrument is giving accurate readings over the working range of the analysis.

11.3.3. The linear range concentrations are dependent on the range of the calibration. Recommended standard concentrations for low level TOC analysis are 0.05, 0.10, 0.50, 1.0, and 5.0 ppm respectively. Recommended standard concentrations for regular (higher level) analysis are 0.50, 1.0, 5.0, 10, 25, and 50 ppm respectively.

11.3.3.1. Analyze each of the linear range verification standards and check each result against the true value.

11.3.3.2. A least squares linear regression is performed on mass:area pairs (see Appendix B). From the slope of the regression line a response factor is calculated as ug C per thousand area counts. A correlation coefficient is also calculated and must be **≥0.995**. The carbon mass from the reagent water is determined from the y-intercept of the regression line.

11.3.3.3. If the results indicate a non-linear response over the range, corrective action is necessary. This may include maintenance and/or recalibration. Maintain documentation of the linear range verification.

11.3.4. An ICV is analyzed following the initial calibration prior to sample analysis. Recovery must be **90-110%** of the true value.

11.3.5. A CCV must be analyzed following every tenth injection and at the end of the run. The CCV is a 25.0 ppm TOC Standard made from stock KHP solution (see 8.2). Recovery must be **90-110%** of the value (**91-106% for Arizona** samples). For low level analyses (i.e. 0.1ppm MRL), the CCV is a 5.0 ppm standard. Calculate the CCV recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Measured concentration of the CCV
TV = True value of CCV

11.3.6. A CCB must be analyzed following every CCV. The CCB is D.I. water, and the result must be below the MRL.

11.4. Sample Analysis

11.4.1. Once system configurations have been established and baseline is stable, the instrument is ready for analysis.

11.4.2. Reagent blank counts must be between 50 and 500 counts. The last 3 counts must be within 50 counts of each other.

11.4.3. Load samples into Autosampler vials and arrange them according to the analytical run sequence shown below. Samples containing suspended solids must be thoroughly mixed prior to sampling.

11.4.4. Analytical Run Sequence. Click Start on the model 1010 to begin analysis. Analyze samples in a analysis sequence as listed below.

11.4.5. When performing method 5310C, analyze all samples in duplicate. The measurements must be within ±10%. If not, repeat the analysis until consecutive measurements are obtained that are reproducible to within ±10%. Since this is an analytical step required to generate the reported result, this also applies to PT samples

11.4.6. When performing method 9060A, analyze all samples in quadruplicate. Since this is a analytical step required to generate the reported result, this also applies to PT samples.

Step	Sample
1	ICV
2	ICB
3	CCV-1
4	CCB-1
5	Method blank
6	LCS
7	Sample
8	Sample-Dup
9	Sample-Spk
10	Rinse blank
11	Rinse blank
12	Sample
13	Sample
14	Sample
15	CCV-2
16	CCB-2

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The ability of each analyst/instrument to generate acceptable accuracy and precision must validated and documented before analysis of samples begins, or whenever significant changes to the procedures have been made To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed. Method criteria must be met for these results.

12.2. Method Detection Limits and Method Reporting Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the ALS *SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (CE-QA011).* The MDL study must be verified annually.

12.3. Limits of Quantification (LOQ)

12.3.1.1. The laboratory must establish a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within 85-115% of the true values to verify the data reporting limit. Refer to the ALS SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (CE-QA011).*

12.3.1.2. The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4. Ongoing QC Samples each sample batch (20 or fewer samples) required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QC Samples are:

12.4.1. A laboratory Control Sample (LCS) must be analyzed with each batch of 20 or fewer samples. The LCS is prepared from a standard which is an independent source from the calibration standards. Acceptance criteria are given in Table 2. This statistically derived acceptance limit is subject to change as limits are updated.

Note: When performing Method 9060 analysis, the second source LCS must be analyzed every 15 samples rather than every 20 samples.

Calculate the LCS recovery as follows:

$\%R = X/TV \times 100$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.4.2. A method blank (Deionized Water) must be analyzed with each batch of 20 or fewer samples. The result must be below the MRL.

12.4.3. In addition to analysis replicates that may be required to obtain the sample result, one sample per service request must be analyzed in duplicate or one per 20 samples, whichever is more frequent. The percent RPD for the duplicates must be **$\leq$17%.** This statistically derived acceptance limit is subject to change as limits are updated. For SM 5310C, all duplicates must be within **10%** RPD.

Relative Percent Difference calculation**:**

$$\text{RPD} = \frac{(S - D)}{((S + D)/2)}$$

where: S = Initial sample result
D = Duplicate sample result

12.4.4. Matrix Spikes- One spike sample must be analyzed per service request or one per 20 samples, whichever is more frequent. Spike 50 ul of 5000 ppm KHP stock solution to 10.0 mls of sample. For low level analysis, spike 50 ul of 1000 ppm KHP stock solution to 10.0 mls of sample. Acceptance criteria are given in Table 2. This statistically derived acceptance limit is subject to change as limits are updated.

Note: Method 9060 requires spike and spike duplicate be analyzed every ten samples.

Calculate percent recovery as follows:

$$\text{Matrix Spike Recovery} = \frac{\text{Spiked Sample} - \text{Sample}}{\text{Spike Added}} \times 100$$

13. DATA REDUCTION AND REPORTING

Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

13.1. Preliminary results are reviewed to determine if dilutions are required. Sample information is transferred to an Excel spreadsheet for calculations (see R:\WET\ANALYSES\TOC\DATA). Instrument baseline is determined by taking the average of all Method Blanks, CCB's, and Rinse Blanks (see R:\WET\ANALYSES\TOC\TOC_CBA1.SPD). Sample concentration is corrected by subtracting calculated blank average (CBA) from instrument response. Concentration and sample identification number are highlighted for reporting purposes.

13.2. For 5310C, report the result from a single analysis. For 9060A, report both the average and the range from the quadruplicate analyses.

13.3. It is the operators' responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in procedures section of the SOP. Average, RPD, spike level and spike recovery are entered on spreadsheet (see append. B) for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.4. Reports are generated in the ALS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be

ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5. As an alternative, reports are generated using Excel© templates located in R:\WET\FORMS. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates the saved to R:\WET\WIP.

13.6. Data Review and Assessment

13.6.1. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action (CE-QA008)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc).
- Sample preservation or handling discrepancies due to laboratory or operations error.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limits Studies and Establishing Limits of Detection and*

Quantitation, (*CE-QA011*). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

17. TRAINING

17.1. Training Outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following *ADM-TRAIN, ALS-Kelso Training Procedure.*

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

19.1. U.S. Environmental Protection Agency, Total Organic Carbon, Method 9060A, Revision 1 November 2004.

19.2. Total Organic Carbon, Combustion-Infrared Method, and 5310C. Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

20. CHANGES SINCE THE LAST REVISION

20.1. Reformatted SOP to current ALS format.
20.2. Added standard phrasing for LOQ in the QA section.
20.3. Added TOC Fusion instrument in the Equipment section.

TABLE 1

Summary of Corrective Actions

Method Reference	**Control**	**Specification and Frequency**	**Acceptance Criteria**	**Corrective Action**
SM 5310C 9060	Linearity verification	Annually	R2 ≥ 0.995	Correct problem then repeat ICAL
SM 5310C 9060	ICV	After ICAL, prior to sample analysis	90-110%	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat initial calibration.
SM 5310C 9060	CCV	Prior to sample analysis, every 10 injections and end	± 10% Diff	Correct problem then repeat CCV or repeat ICAL
SM 5310C 9060	Method Blank	Include with each analysis batch (up to 20 samples)	<MRL	If target exceeds MRL, reanalyze to determine if instrument was cause. If still noncompliant then: Re-extract or reanalyze samples containing contaminate, unless samples contain > 20x amount in blank.
SM 5310C	Laboratory Control Sample	Include with each analysis batch (up to 20 samples)	See DQO	If exceeds limits, re-extract and re-analyze
9060	Laboratory Control Sample	Include with each analysis batch (up to 15 samples)	See DQO	If exceeds limits, re-extract and re-analyze
SM 5310C	Matrix Spike	Include with each analysis batch (up to 20 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
9060	Matrix Spike	Include with each analysis batch (up to 10 samples)	See DQO	Evaluate data to determine if the there is a matrix effect or analytical error
SM 5310C	Sample Duplicates	All samples in batch	≤ 10 % RPD	Re-homogenize and re-analyze if result is > 5 X the MRL
9060	Sample Quadruplicates	All samples in batch	≤ 17 % RSD	Re-homogenize and re-analyze if result is > 5 X the MRL

TABLE 2

METHOD	**ANALYTE**	**MATRIX**	**MDL**[a]	**MRL**	**LOD**[b]	**LOQ**[c]	**UNITS**
9060A	Total Organic Carbon	Water	0.07	0.5	0.2	0.5	mg/L
SM5310 C	Total Organic Carbon	Water	0.08	0.5	0.16	0.5	mg/L
SM5310 C LL	Total Organic Carbon	Water	0.04	0.1			mg/L

a Method Detection Limits are subject to change as new MDL studies are completed.
a MDL is the smallest analyte concentration that can be demonstrated to be different from zero with 99% confidence
b The LOD is the smallest amount of a substance that must be present in a sample in order to be detected with 99% confidence. Verification is acceptable if the response is > 3x instrument noise.
c The LOQ is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.

ALS Standard Operating Procedure

DOCUMENT TITLE:	*ANALYSIS OF TOTAL ORGANIC CARBON*
REFERENCED METHOD:	*EPA 415.1, SW9060 A, SM5310 C*
SOP ID:	*670*
REV. NUMBER:	*14*
EFFECTIVE DATE:	*AUGUST 5, 2011*

ALS
STANDARD OPERATING PROCEDURE 670 REVISION 14

TITLE: ANALYSIS OF TOTAL ORGANIC CARBON BY METHODS EPA 415.1, SW9060A AND SM5310 C

APPROVED BY:

PRIMARY AUTHOR____________________________DATE __________

QUALITY ASSURANCE MANAGER ______________________DATE __________

LABORATORY MANAGER ________________________ DATE __________

In order to keep personal signatures secure, this document in electronic form has been formatted and approved for use in accordance with ALS SOP 926 “Controlled Document Management” and all printed copies are uncontrolled documents. Signatures are on file

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- EPA 415.1, SW9060A and SM5310 C -- describe procedures for the analysis of Total Organic Carbon (TOC) in water. These procedures are applicable to the measurement of organic carbon contained in drinking, surface, ground, and saline waters, as well as domestic and industrial wastes. Exclusions are noted under Interferences (Section 4).

This procedure is applicable only to homogenous samples that can be injected into the instrument reproducibly by the autosampler.

The forms of carbon that can be measured by this procedure include the following:

- Soluble, nonvolatile organic carbon (e.g., natural sugars)
- Soluble, non-purgeable volatile organic carbon (e.g., mercaptans, alkanes, low molecular weight alcohols)
- Insoluble, partially volatile carbon (e.g., low molecular weight oils)
- Insoluble, particulate carbonaceous materials (e.g., cellulose fibers)
- Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter (e.g., oily matter adsorbed on silt particles).

Because of purging, most volatile organic solvents may be lost.

2. SUMMARY

TOC concentration in water is measured by the use of an automated TOC analyzer. The sample is acidified (if not preserved prior to receipt) and sparged with nitrogen (N_2) gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide (CO_2) by persulfate ($S_2O_8^{-2}$) in the presence of ultraviolet (UV) light. The resultant CO_2 is sparged from the sample and carried in a stream of N_2 gas to a non-dispersive infrared detector (NDIR). TOC concentration in the sample is calculated as a function of CO_2 peak area by use of a linear equation generated from a previously analyzed multipoint initial calibration. Sample aliquots, reagents and waste are transferred through the system by means of the autosampler apparatus.

Dissolved Organic Carbon (DOC) can also be measured by this procedure. Although ALSLG-FC prefers that samples be filtered prior to receipt at the laboratory, this filtering can be done after receipt.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALSLG-FC's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALSLG-FC standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

3.6 When a specific work order is designated as "QSM Compliance", the criteria specified in ALS SOP 996 shall be followed. Additionally any client requirement shall also be followed. Specific criteria noted in this SOP are superseded"

4. INTERFERENCES

4.1 Any inorganic carbon (e.g., dissolved CO_2, HCO_3^-, and CO_3^{-2}) present in the sample at the oxidation step will contribute to the CO_2 reaching the detector and consequently give a high bias to the measured TOC concentration. Inorganic carbon must either be removed from the sample prior to the oxidation step, or be accounted for in the final calculation. When the Phoenix 8000 instrument is operating in the TOC mode, the sample is routinely acidified and sparged to remove inorganic carbon prior to oxidation of organic carbon. Note that volatile organic compounds may be lost when inorganic carbon is sparged from the sample.

4.2 A study published by the instrument vendor (Tekmar-Dohrmann) indicates that sulfuric acid (H_2SO_4) could form SO_3 gas in the UV reaction cell. Because SO_3 has similar absorption in the infrared region as CO_2, the SO_3 can cause a positive interference in the NDIR detector of the instrument. Therefore, it is recommended that phosphoric acid (H_3PO_4) be used instead of H_2SO_4 where acid preservation is designated for aqueous TOC samples.

Acidification to pH$\leq$2 at time of collection is desirable for unstable samples, however, it should be noted that acid preservation invalidates any inorganic carbon determination on the samples.

4.3 Chloride (Cl^-) ions can react with persulfate in the reaction cell to form Cl_2 (gas). If the Cl^- concentration in a sample is high ($\geq$ 1000mg/L) this reaction can compete with the oxidation of organic C for persulfate. This reaction can lead to excessive peak tailing of the signal from the NDIR detector. At very high Cl^- concentrations (common to brines, seawater, and some chemical wastewaters) the effect can be severe and low TOC recovery can result because some of the organic matter will not be oxidized in the established analysis time. Therefore, hydrochloric acid (HCl) should not be used as a preservative for water samples designated for TOC analysis. As noted previously, the instrument manufacturer recommends the use of phosphoric acid as a preservative for aqueous samples.

4.4 Because solid particles can plug or damage the 8-port valve in the instrument, it may be necessary to filter samples that contain particulates or to allow the solids to settle out prior to analysis.

5. APPARATUS AND MATERIALS

5.1 Phoenix 8000 TOC analyzer (Tekmar-Dohrmann), or equivalent

5.2 pH paper, narrow-range, acidic

5.3 Vials, glass, 40mL VOA-type

5.4 Syringe filters, Life Sciences IC Acrodisc®, 25mm, 0.45um Supor® (PES) membrane, or equivalent, for filtering samples prior to DOC analysis (Section 12)

6. REAGENTS AND STANDARDS

Refer to ALS SOP 300 “Standards, Solvents, Acid, Bases and Reagents Management in the Laboratory”

6.1 Nitrogen (N_2), 99.999% purity, used as carrier and purge gas

6.2 Reagent water, (HPLC grade or Milli-Q ASTM Type II)

6.3 Phosphoric acid, H_3PO_4, concentrated, reagent grade

6.4 Acid reagent for IC sparging: Add 100mL conc. H_3PO_4, to 500mL of reagent water.

6.5 Potassium hydrogen phthalate (KHP), used to create the in-house first-source TOC stock solution.

6.6 Copper (Cu) granules

6.7 Tin (Sn) granules

6.8 Sodium persulfate reagent: transfer 100g of sodium persulfate ($Na_2S_2O_8$) to a large beaker. To the beaker add 850mL of reagent water and 36mL of conc. H_3PO_4. Place a magnetic stir bar into the beaker and stir on a magnetic stir plate until all of the solid particles are dissolved. (expiration date = 1 year).

6.9 STANDARDS

6.9.1 All standards are maintained per SOP 300. In the event of a conflict, the specific guidance in this SOP will supersede that of SOP 300.

6.9.2 TOC stock solution, 1000mg/L TOC, first source: Prepared in-house by adding 2.13g of KHP ($C_8H_5KO_4$) to a 1L Class A volumetric flask half-filled with reagent water. Place a magnetic stir bar into the flask and stir on a magnetic stir plate until all of the solid particles are dissolved. Carefully add 1.0mL of phosphoric acid to acidify the solution to pH ≤ 2, let cool to room temperature. Bring to near full volume with reagent water and verify solution pH as ≤ 2. Bring to full volume with reagent water. **Refrigerate.** The expiration date of this solution is 1 year or less as described in SOP 300. Discard the solution if a precipitate forms or degradation is suspected.

6.9.3 Initial calibration standards: Prepared at a minimum of 5 levels to bracket the linear range of the detector. Prepared by diluting aliquots of the 1000mg/L TOC stock solution with reagent water. Calibration standards with concentrations of 10mg/L or greater can be stored for 1 year or as described in SOP 300. Standards with concentrations of less than 10mg/L are made daily upon use.

6.9.4 “Demand” TOC reference standard, second source: This is a stock standard solution obtained from a commercial vendor that is used to prepare the ICV/LCS standard. Alternately, the standard can be prepared in-house from sources independent of the calibration solutions, per the directions contained in the referenced method. The expiration date of this standard is the manufacturer’s expiration date or 1 year from preparation ($\geq$10mg/L), whichever is shorter.

6.9.5 ICV/LCS (Initial Calibration Verification and Laboratory Control Sample): An aliquot of the "Demand" TOC reference stock standard is diluted with reagent water according to instructions provided by the vendor. The reference concentration of the prepared standard is provided by the vendor and may vary from lot number to lot number. The concentration of the ICV is typically different from the CCV and between 20-40mg/L.

6.9.6 CCV (Continuing Calibration Verification) standard: An aliquot of the TOC stock solution is diluted with reagent water to a concentration at or below the mid-point of the calibration range. The concentration of the CCV is typically 30mg/L for a calibration range of 0.5-60mg/L. This standard expires in the shorter of 6 months or the expiration date of the standard it was prepared from.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 All samples should be collected according to an approved sampling plan. ALS SOP 202 "Login and Distribution of Samples and Workorders" and ALS SOP 205 "Preparation of Bottle Orders, Shipping Sample Kits, Maintaining Inventory of Bottles, Preservatives and Labels" provide guidance for this subject.

7.2 Sampling and storage of samples in amber glass bottles is preferable. Plastic containers, such as conventional polyethylene and cubitainers, are permissible if it is established that the containers do not contribute contaminating organics to the sample or adsorb organics from the sample.

7.3 Methods EPA 415.1 and SW9060A provide for chemical preservation of samples using either hydrochloric (HCl) or sulfuric (H_2SO_4) acid. Method SM5310 C provides for chemical preservation of samples using either sulfuric or phosphoric acid (H_3PO_4). As discussed in Section 4.2, a technical note released by the instrument manufacturer (Tekmar-Dohrmann) recommends use of phosphoric acid to avoid possible instrumental interferences. Although ALSLG-FC can accept and process samples preserved with any of the three acids, it is ALSLG-FC's preference and practice to provide for phosphoric acid preservation to $pH \leq 2$.

7.4 The referenced methods do not prescribe a maximum holding time allowance. Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between collection of samples and analysis should be minimized. ALSLG-FC's policy is to analyze samples within 28 days of collection.

7.5 Samples should be kept cool (4±2°C) and protected from sunlight and atmospheric oxygen.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 INSTRUMENT SET UP

Prior to analyis, check to see each of the following are adequate for the amount of samples to be analyzed:

8.1.1 N_2 carrier gas, 500^{+}psi from cylinder.

8.1.2 Ample supplies of persulfate reagent, sparging acid, and reagent water.

8.1.3 Halogen scrubber, ample life.

8.1.4 Carrier gas flow rate (200cc/min, ±10%).

8.1.5 Gas/liquid separator water level filled to waste outlet.

8.1.6 Mist trap is empty, drain if necessary.

8.1.7 Thumbscrews of 8-port valve are hand tightened.

8.2 INITIAL CALIBRATION

8.2.1 Prepare calibration standards as described in Section 6.9 above. Typical concentrations comprising the calibration curve are 1.0, 4.0, 10, 20 and 40ppm.

8.2.2 Analyze the calibration standards on the instrument using the instrument software (TOC Talk™).

8.2.3 After analyzing the standards, the instrument software will calculate a linear equation to fit concentration with instrument response. To be acceptable, the coefficient of variation (r^2 or “r-squared” value on the output) must be 0.99 or greater.

8.3 CALIBRATION VERIFICATION

8.3.1 ICV: After an acceptable initial calibration has been established, an initial calibration verification (ICV) check standard must be analyzed. The ICV must be prepared from a parent source that is independent from that used to prepare the calibration standards. The ICV is typically prepared at a concentration near the midpoint of the calibration range, although other concentrations should be analyzed occasionally. See Section 6.9.4 above for preparation guidance, and QC Table following for acceptance criteria and corrective measures to be taken if necessary.

Since there is no sample preparation step involved in this analysis, the ICV check standard can serve a dual role as the laboratory control sample (LCS) for a quality control (QC) batch of 20 or fewer samples.

8.3.2 CCV: A CCV check standard is run at the beginning and conclusion of each analytical sequence and after every 10 samples in the sequence. **If running samples by SW9060A protocol, this CCV should be prepared from a source other than that used to prepare the ICAL (i.e., a second**

source). Preparation of the CCV is described in Section 6.9.5. Refer to QC Table following for acceptance criteria and corrective measures to be taken if necessary.

8.4 SAMPLE ANALYSIS

8.4.1 Samples must be analyzed for TOC in QC batches of 20 or fewer samples. See Section 9 for QC requirements (type and frequency). Confirm that pH is $\leq$2 for each sample prior to analysis and record the pH test result.

8.4.2 Prior to aliquoting, all samples should be homogenized by thorough shaking or agitation of the sample bottle.

8.4.3 For samples analyzed per Method SW9060A protocol, quadruplicate analyses must be performed for all field samples. Report the average result of the four (4) analyses and the RSD (Relative Standard Deviation). The range of values may be obtained from the raw data.

8.4.4 If the TOC concentration of a sample exceeds the calibration range (i.e., exceeds the concentration of the highest calibration standard), the sample must be diluted and reanalyzed as necessary until the concentration is within range.

9. QUALITY CONTROL (QC)

See QC Table following for acceptance criteria and corrective measures to be taken if necessary. Refer to Quality Control Samples defined in QAM section 14.9.

9.1 METHOD BLANK

One method blank (MB) must be analyzed with every QC batch of 20 or fewer samples to demonstrate that potential contaminants within the analytical system are in control. The MB consists of an aliquot of reagent water.

9.2 LABORATORY CONTROL SAMPLES

One laboratory control sample (LCS) must be analyzed with every QC batch of 20 or fewer samples to demonstrate the effectiveness of the analytical system. The LCS composition is identical to that of the ICV check standard (see Section 6.9.4). Since there is no preparation step in this analysis, the ICV check standard at the beginning of an analytical sequence can serve a dual role as the LCS for a QC batch.

9.3 MATRIX SPIKES

Matrix spike (MS) samples consist of field samples into which known concentrations of target analytes have been introduced. Analysis of matrix spikes provides information on the effect of sample matrix on target analyte detection. A matrix spike duplicate (MSD) is typically run with the MS.

Sample volume permitting, one pair of matrix spike/matrix spike duplicate (MS/MSD) analyses must be performed for every 20 samples. The matrix spiked samples are prepared by spiking aliquots of a selected field sample in the preparation batch with aliquots of the 1000mg/L stock standard.

Analyte recovery for the MS and MSD is calculated as shown below:

$$\%R = \frac{(\text{Conc.}_{\text{Found}} - \text{Conc.}_{\text{.Sample}})}{\text{Conc.}_{\text{Target}}} \quad X \quad 100$$

where:

$\text{Conc}_{\text{Found}}$ = analyte concentration found in the MS or MSD sample
$\text{Conc}_{\text{Sample}}$ = analyte concentration found in the field sample
$\text{Conc}_{\text{Target}}$ = target (anticipated) analyte concentration based on amount spiked

As a measure of precision, the relative percent difference (RPD) of the laboratory duplicate sample pair (or MS/MSD or LCS/LCSD pair) is calculated as shown below:

$$\text{RPD } (\%) = \frac{(\text{Result}_{\text{MS}} - \text{Result}_{\text{MSD}})}{(\text{Result}_{\text{MS}} + \text{Result}_{\text{MSD}}) / 2} \quad X \quad 100$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. The LCS, MS, or both may be analyzed in duplicate to serve this purpose. Precision is expressed as Relative Percent Difference (RPD) (see above).

SW9060A protocol requires a “spike duplicate sample for every 10 samples”. If analyzing samples by SW9060A protocol, include either an LCSD or (if sufficient sample volume is provided) an MSD *for every 10 samples analyzed*. If there is insufficient sample for the MSD, then either a second LCS/D pair can be analyzed in the latter half of the prep batch, or prep batches may be limited to 10 samples. **Note that this requirement does not apply to samples being analyzed by Method 415.1.**

9.5 LOD/LOQ

See ALS SOP 329 for LOD/LOQ/detection limit determinations..

10. DEVIATIONS FROM METHOD

See discussion in Sections 4.2 and 7.4 regarding acid preservation of samples. Methods 415.1 and SW9060A both describe the homogenization of samples by means of a blender. In order to protect the instrument from being clogged by particulate matter, this approach is not utilized at ALSLG-FC (see Section 8.4). This SOP contains no other known deviations from the promulgated methods.

11. CORRECTIVE ACTIONS AND CONTINGENCIES

REFER TO ALS SOP 928 “NON-CONFORMANCE AND CORRECTIVE ACTION PROCEDURES

12. SAFETY, HAZARDS AND WASTE DISPOSAL

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP

11.1 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP) .

13. DEFINITIONS

Definitions are listed in QAM appendix A "Glossary."

14. REFERENCES

12.1 USEPA, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, Method 415.1, "Total Organic Carbon by Combustion or Oxidation", 1983.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 9060A", Revision 1, November 2004.

12.3 Standard Methods for the Examination of Water and Wastewater, 20th Ed., 1999. "Total Organic Carbon, Persulfate-Ultraviolet Method", 5310 C.

12.4 Phoenix 8000 User Manual, Tekmar-Dohrmann, 1998.

12.5 Application Note, "TOC Analysis: The Acid Preservation Debate", Tekmar-Dohrmann, 2001.

12.6 "Method Development Study: Dissolved Organic Carbon (DOC)", Darryl Patrick, 2007. J:\QAOffice\Demonstrations\

Analytical Method: EPA 415.1; SW9060A, SM5310 C	**Parameter:** Total Organic Carbon (TOC) by Oxidation		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria **	Corrective Action
Initial Calibration, minimum 5-point	As needed (i.e., at on-set of analyses or when continuing calibration does not meet criteria)	r^2 must be ≥ 0.99	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Initial Calibration Verification (ICV), second source check standard run near mid-point of calibration curve (Because no sample preparation steps are involved, the ICV can also serve as the LCS for the initial QC batch of samples analyzed)	Once after each initial calibration	For Method 415.1 and SW9060A analyses, the ICV result must be within ±15% of the expected concentration	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV), run at or below midpoint of calibration; CCV concentration must be different from ICV concentration	Run after every 10 samples to begin and end an analytical sequence	For Method 415.1 and SW9060A analyses, the CCV result must agree within ±15% of the expected concentration	Check that calculations and preparation are correct, evaluate/ correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.
Laboratory Control Sample (LCS), second source standard run near mid-point of calibration curve (The ICV can also serve as the LCS for the initial QC batch of samples analyzed)	One LCS in every QC batch of 20 or fewer samples	For Method 415.1 and SW9060A analyses, the LCS result must be within ±15% of the expected concentration	Check calculations, spike preparation, and freshness of the standard used for spiking. Prepare another LCS and analyze. If LCS still fails, samples in QC batch must be reanalyzed.
Laboratory Duplicate (DUP)	For Method 415.1 and SW9060A, the LCSD & MSD both can serve as a laboratory duplicate analysis	For both Method 415.1 and SW9060A, the RPD between the duplicate pair should be $\leq$20%	For RPDs outside of QC limits, check all calculations for errors. Narrate.
Method Blank (MB)	One MB per every QC batch of 20 or fewer samples	For Method 415.1 and SW9060A analyses, the MB result must not exceed RL (usually 1mg/L TOC)	Prepare another MB and analyze. If MB still fails, samples in QC batch must be reanalyzed.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	Volume permitting, one MS/MSD pair per batch of $\leqq$ 20 field samples	For Method 415.1 and SW9060A analyses, MS/MSD recoveries should meet advisory limits of ±20% (80-120% of the expected values) and RPD should be $\leq$ 20	Check for documentable errors (e.g., calculations and spike preparation). For Method 415.1 and SW9060A analyses, sample matrix effects are the most likely cause if no errors are found. Document and note in case narrative.

Analytical Method: EPA 415.1; SW9060A, SM5310 C	**Parameter:** Total Organic Carbon (TOC) by Oxidation		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria **	Corrective Action
Method Detection Limit (MDL) Study; run per guidance in SOP329	As needed and, at minimum, annually	Positive result < analyte reporting limit (usually 1.0PPM for both Method 415.1 and SW9060A analyses)	Determine the reason for failure and correct problem with system; then repeat study. If MDL study still not acceptable, discuss with Department and QA Managers, RL may be adjusted, if necessary.

**** Acceptance Limits are as stated within Table, or as otherwise specified in the applicable LIMS program specification.**

Appendix A

Preservation/Acidification and Filtration of Water Samples for TOC/DOC **SOP 670 rev. __** ***ALS Laboratory Group***

Workorder ID / Sample ID	DOC							TOC / DOC			Comments
	Date	Initials	Samples Filtered by PAI or Client	Filtered Through 0.45um? (Y/N)	Filter Spec's LOT #	Volume of H_3PO_4 Added (mL)	Conc. H_3PO_4 LOT #	Date	Initials	pH at Time of Analysis	

Filter Blanks = Milli Q Double Deionized Water.

Reviewed by/date ______________________

Instrument SN 01-011007

Form
647r1.xls

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF DISSOLVED GASES IN WATER SAMPLES USING GAS CHROMATOGRAPHY
REFERENCED METHOD:	RSK 175
SOP ID:	449
REV. NUMBER:	3
EFFECTIVE DATE:	AUGUST 10, 2018

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 449 REVISION 3

TITLE: **DETERMINATION OF DISSOLVED GASES IN WATER SAMPLES USING GAS CHROMATOGRAPHY**

FORMS: **NONE**

APPROVED BY:

PRIMARY AUTHOR ____________________ DATE ____________

QUALITY ASSURANCE MANAGER ____________________ DATE ____________

LABORATORY MANAGER____________________ DATE ____________

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references – RSKSOP-175 and EPA Region I Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, Ethene, and Propane, is used to determine concentration of dissolved gases (methane, ethane and ethane) in water samples.

Analyte	CAS #	Molecular Weight
CH_4	74-82-8	16
$C2H_4$	74-85-1	28
$C2H_6$	74-84-0	30
$C3H_8$	74-98-6	44

Other compounds may be analyzed if successful demonstration of capability (DOC) and method detection limit (MDL) studies are performed.

2. SUMMARY

A headspace volume is created at ambient pressure in each water matrix blank, calibration standard, sample and quality control sample.. Equilibrium is then attained by vortex mixing (or equivalent equilibration), and an aliquot from the headspace is then introduced to a gas chromatograph with flame ionization detection (GC/FID), to determine the concentration of dissolved gases in the water sample.

Every water blank, calibration standard, sample and quality control (QC) sample is treated equivalently. The temperature, pressure, headspace volume, equilibration procedure and injection volume are kept constant.

Standards are prepared by introduction of a selected volume of gas phase standard to the headspace of a laboratory reagent blank. The standard concentrations are calculated to reflect the total concentration (TC) of analyte per volume of water. After equilibration, a portion of the headspace in each standard is analyzed, and a linear or 2nd order calibration curve is generated that describes the relationship between instrument response and standard concentration.

The TC of each dissolved gas in each water sample is then determined by analysis and comparison of the resulting instrument response to the calibration curve.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

3.6 If the words “QSM Criteria” appear on the WIP and Tracking Sheets for a specific work order, that work order requires the criteria as specified in Appendices B and C of SOP 996, or as defined in the appropriate Program Specification.

3.7

4. INTERFERENCES

4.1 Any co-eluting entity that responds via FID. Few method interferences are known because methane, ethene and ethane are very small and highly volatile molecules. Interfering compounds are likely to be much more highly retained on the analytical column and separated from these analytes. Typically, chromatographic interferences are not observed with this procedure.

4.2 Interferences are also minimized by the use of high purity reagents (helium or nitrogen, analyte-free water).

4.3 Methane, ethene, ethane, and propane may be present in the atmosphere or from a source that produces contamination (methane more so than ethene, ethane, or propane). Precautions should be taken to ensure that interference in room air is avoided. Refer to QC Table for guidance and corrective actions pertaining to method blank analyses.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH (GC) AND DETECTORS

Hewlett Packard 5890 Series II GC or equivalent equipped with a flame ionization detector (FID)

5.2 DATA ACQUISITION

Any data acquisition system capable of acquiring, storing and processing GC/FID data (e.g. Agilent EZChrome^{TM} or equivalent) to support the qualitative and quantitative requirements of this method may be used.

5.3 GASES - use only ultra high purity (+/- 2%)

Helium (purge and carrier gas)

Hydrogen (FID detector gas)
Compressed Air (FID detector gas)

5.4 COLUMNS - Equivalent columns may also be used
Analytical Column: J&W GS-CARBONPLOT; 30m x 0.533mm x 3.00µm;
0-360ºC operating range

5.5 MEASURING DEVICES
Gas Tight Syringes, various µL ranges

5.6 CONSUMABLES

- GC septa
- VOA Vials, 40mL size
- Replacement caps with septa, for 40mL VOA vials

5.7 Vortex mixer

6. REAGENTS AND STANDARDS

6.1 GAS PHASE STANDARDS:

6.1.1 Air Liquide-Scott Specialty Gases^{TM}: Scotty® mix; methane, ethane, ethane at various applicable concentration such as 100ppm (mole), 1% (mole) and 30% (mole) in nitrogen, or equivalent

6.1.2 Alternate source check standard, if available, otherwise two different lots may be used to confirm accuracy (ICV). Other reference material concentrations may be used, as long as an appropriate calibration range is accomplished.

6.2 Organic-free reagent water; carbon-filtered, boiled and purged with helium prior to use (SOP 511)

6.3 Methanol, HPLC grade

6.4 STANDARDS

6.4.1 All standards are maintained per SOP 300. Specific SOP instructions take precedence with regard to management of standards.

6.4.2 At minimum, two independent sources of target analyte are recommended. First source materials are used to create calibration, continuing calibration verification (CCV) and QC sample spike standards. Second source materials are used to create the initial calibration verification (ICV), which is used to independently verify the accuracy of the initial calibration (ICAL). Use a second certified standard lot if a suitable alternate standard supply is not available.

6.4.3 An appropriate volume of stock standard is aliquoted to create working standards. All standards are delivered using gas tight syringes or a vacuum manifold system or other accurate gas delivery technique. Standards diluted from stock should be prepared daily.

6.4.4 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

Samples should be collected according to an approved sampling plan. Refer to ALS SOP 202 “Login and Distribution of Samples and Workorders” and ALS SOP 205 “Preparation of Bottle Orders, Shipping Sample Kits, Maintaining Inventory of Bottles, Preservatives and Labels

7.1 Samples should be acidified with hydrochloric acid (HCl) to pH < 2. Water samples are usually preserved by adding approximately four (4) drops of concentrated hydrochloric acid (HCl) to each 40mL VOA vial. The purpose of the hydrochloric acid is to prevent microbially induced bias of target compound concentration. If the water sample is unpreserved, the holding time is not defined. Sample analysis of unpreserved samples may proceed given client approval.

7.2 Aqueous samples are collected in 40mL glass VOA vials with screw tops and Teflon TM-lined septa. Aqueous samples should be headspace-free. It is recommended that a minimum of three vials should be collected for each field sample. For a designated matrix spiked (MS) analysis, the client may need to provide as many as six vials. Note that a matrix spiked duplicate (MSD) analysis is not typically performed with this procedure.

7.3 Store samples at 4±2 ° C

7.4 Samples must be analyzed within 14 days of sample collection.

7.5 To prevent loss of volatile organic compounds, samples must not be opened until the time of analysis.

7.6 Refer to ALS SOP 336 Representative Laboratory Subsampling – Stable Chemistry for soil samples, as applicable

8. PROCEDURES

8.1 TYPICAL SYSTEM OPERATING CONDITIONS

Gas Chromatograph

Column Flow Rate (helium):	10.0 ± 0.5mL/min
Air Flow Rate	per manufacturer's recommendation
Hydrogen Flow Rate:	per manufacturer's recommendation
Purge Valve:	On
Injector Temp.	250°C
Detector Temp	320º C
Column Temperature:	150°C
[1]Ramp:	150°C, 4min., 40°C/min., 240°C, 0.8min.
[1]Run Time:	8 min. (includes ramp
Injection Volume:	300 µL

[1] The run time may be shortened by omitting the ramp. The shortened run is then an isothermal run. Periodic bake out or use of the temperature ramp may be important to avoid carryover effects in some sample matrices.

8.2 GC MAINTENANCE

Prior to establishing calibration curve or analyzing samples, the following suggested maintenance can be performed to aid in achieving more consistent results:

- Change the GC injection port septum regularly (after approximately each 50 injections).

- Bake out the GC at 250°C until the background signal reaches approximately 4 mV.

- Clean or change the GC liner if pieces of septa or other contamination begin to cause a rise in background signal or column bleed.

- Syringes may be purged with helium or nitrogen to control potential carryover effects.

- Additional GC bake-out may be added to routine sample runs to control moisture or late eluting interferences.

8.3 DISSOLVED GASES CONCEPTS

The purpose of this procedure is to identify and quantitate the concentration of a dissolved gas (methane, ethene, ethane, or propane) in an aqueous field sample.

8.3.1 Starting with a 40mL VOA vial (42.5 mL of volume), a 4.0mL headspace is created.. Thus, 38.5mL of water sample remains.

At this point, any target analytes in the water partition into the headspace until equilibrium between the two phases is reached.

The concentration of target analyte in the original sample can be said to be equal to the mass of analyte partitioned to the headspace plus that remaining in the water, divided by the 38.5mL of water sample remaining in the vial.

8.3.2 A standard can be prepared in the same manner as the sample. 4.0mL of headspace is created in a vial of blank reagent water. A known amount of a reference gas standard is then added to the headspace (an equivalent amount of headspace is first withdrawn to maintain ambient pressure inside the vial), and the standard is allowed to equilibrate (same conditions as a field sample). The resulting concentration can be defined as the total mass of analyte added, divided by the water volume in the vial (38.5mL).

If a series of initial calibration standards (at different concentrations) is thusly prepared and analyzed, a calibration curve may be generated from the detector responses obtained.

Field samples may then be analyzed and their detector responses compared to the calibration curve for quantitation.

Since water volume, headspace volume, total VOA vial volume, equilibration conditions, pressure and temperature are all kept constant between standards and samples, one may calibrate and quantitate without the need to determine the concentration in the water phase using the Henry's law calculation approach.

Reference gas standards are supplied with the analyte concentration stated in ppm as calculated on a mole basis. It is necessary to first calculate the weight per unit volume concentration of each analyte in each standard. This concentration is then used to calculate the total mass of analyte added to a standard or QC sample (via addition into the headspace).

8.3.3 CALCULATIONS

A. Unit Conversion of ppm (mole basis) to gram/liter for Gas Mixtures:

Example Given a 100 ppm (mole basis) gas mixture of CH_4 in nitrogen, calculate the concentration (g/L) of CH_4 in a 1 liter volume:

Key Assumptions

- Temperature of sample assumed to be at 22°C

- Pressure of sample assumed to be 840 bar (0.829 atm; typical Fort Collins atmospheric pressure) just before injection.

  At around atmospheric pressure, gases behave in close to ideal manner.

Using the Ideal Gas Law (PV = nRT) for a temperature of 295.15°K (22°C), a pressure of 0.829 atm (= 840mbar barometric pressure), and the gas constant R of 0.0821 liter-atm/mole-°K, it is determined that:

1 mole of ideal gas occupies 29.21 liters.

One liter of gas will then contain (1/29.21) moles.

Since the concentration of CH_4 is 100 ppm:

(total # moles per liter)(concentration of CH_4) = total number of moles of CH_4 in 1L

The concentration of 100 ppm (parts per million) is unit-less, and equals 100 mole-parts per 1,000,000 total moles = 0.000100 in decimal form; thus the amount of moles of CH4 in one liter of mixture is:

(1/29.21 moles/L)(0.000100) = 0.00000342 moles of CH_4 per liter

The analyte's molecular weight is used to determine the weight of analyte in the mixture:

Example For methane (molecular weight 16 gram/mole):

(16 gram/mole)(0.00000342 moles/L) = 0.0000547 g/L or 0.0547 mg/L

B. General Formula for Conversion of ppm (mole) to gram/liter for Gas Mixture (22°C, 0.829 atm):

$$\frac{\text{Gas Conc. (ppm in decimal form) x mole-weight (gram/mole)}}{29.21\ \text{L / mole}} = \text{Conc. (g/L)}$$

C. Calculation of Concentration for Standards:

$$\frac{\text{Gas Conc. (ppm in decimal form) x mole-weight (g/mole) x Vol. Std. Added to Headspace (L)}}{\text{Volume at ambient P and T (L/mole) x Vol. Sample (L)}} = \text{Conc. (mg/L)}$$

Unit Conversions: mg/L (1000 µg/mg) = µg/L

NOTE: The gas volume to be added is first withdrawn from the headspace to maintain ambient pressure (equivalent headspace volume and pressure to that of the samples).

Concentration = total mass of analyte (in the entire vial)/volume of water in vial, mg/L or µg/L

MW = Molecular weight of analyte (g/mole)

T = Temperature

P = Pressure

Example Calculation Standard Concentration for methane (injection of 4µL of 1% mole/mole standard into a VOA vial with 4.0mL headspace):

$$\frac{(0.0100\text{ mole ratio; 1\% std.})(16\text{g/mole})(4\text{x}10^{-6}\text{L injected})(1\text{x}10^{6}\mu\text{g/g})}{(29.21\text{ L std. Vol. for 1 mole})\ (0.0385\text{ L water})} = 0.569\ \mu\text{gCH}_4/\text{L}$$

Calculation of sample results may be done directly by comparison to the standard total concentration curve.

8.4 INITIAL CALIBRATION

As appropriate, refer to ALS SOP 337 “Organics Calibration Procedures –- Method 8000C”.

8.4.1 Initial calibration standards are prepared at a minimum of five concentrations. The range of concentrations of the initial calibration is intended to define the working range of the analytical system. One of the concentrations must be at or below the analyte reporting limit.

8.4.2 Certified gas standards, containing target analytes in nitrogen, are used to prepare the working standards. The calibration standards are prepared from water blanks and are handled as samples would be (equivalent headspace, pressure, temperature, equilibration).

8.4.3 Example standard levels are provided in the following Table. Standard concentrations were calculated based on a room temperature of 22 ºC and a barometric pressure of 840mbar. As the barometric pressure varies by less than 3% from 840mbar in Fort Collins, CO and room temperature is maintained at close to 22ºC in the laboratory, these conditions will be routinely applied to calculate standard concentrations. Standard concentrations should be corrected if ambient conditions vary significantly.

TABLE 3
CALIBRATION STANDARDS

Target	Standard Preparation	Total Conc. ppm	Total Conc ppb
CH_4	4µL 1%	0.0005690	0.5690
	25µL 1%	0.0035563	3.5563
	100µL 1%	0.0142253	14.2253
	1000 µL 1%	0.1422532	142.2532
	300 µL of 30%	1.282789	1280.2789
	3000µL of 30%	12.8027886	12802.7886
$C2H_4$	4µL 1%	0.0009958	0.9958
	25µL 1%	0.0062236	6.2236
	100µL 1%	0.0248943	24.8943
	1000µL 1%	0.2489431	248.9431
	300µL of 30%	2.2404880	2240.4880
	3000µL of 30%	22.4048801	22404.8801
$C2H_6$	4µL 10%	0.0010669	1.0669
	25µL 1%	0.0066681	6.6681
	100µL 1%	0.0266725	26.6725
	1000µL 1%	0.2667248	266.7248
	300µL of 30%	2.4005229	2400.5229
	3000µL of 30%	24.0052287	24005.2287
$C3H_8$	25µL0 .1%	0.0009781	0.9781
	100µL 0.1%	0.0039126	3.9126
	10µL 5%	0.0195628	19.5628
	50µL 5%	0.0978139	97.8139
	200µL 5%	0.3912554	391.2554
	250µL 5%	0.4890693	489.0693
	1000µL 5%	1.9562772	1956.2772
	2000µL 5%	3.9125544	3912.5544
	500µL 100%	19.5627720	19562.7720

NOTE: Alternate equivalent dilution schemes may be used as appropriate. Five or more calibration levels are required. Sample injection size, headspace volume, water volume, pressure and temperature must be equal for all standards and samples (keep constant throughout each run).

8.4.4 Inject 300µL of each equilibrated calibration standard into the GC and acquire data.

8.4.5 Electronically integrated peak area responses are tabulated and quantitated using external standard quantitation. Calibration Factors (CFs) for each compound are calculated as follows:

$$CF = A_s/C_s$$

where:

A_s = response (area) for the analyte to be measured

C_s = concentration of the analyte to be measured (µg/L)

8.4.6 Since each CF represents the slope of the line between the response for that standard and the origin, then if the observed deviation between the CF's is constant (i.e., ≤20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample concentrations. Percent Relative Standard Deviation (%RSD) is calculated as:

$$\%RSD = \frac{\text{Standard Deviation (SD)} * 100}{\text{Average (mean) RF}}$$

If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

8.4.7 When %RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be >0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. If non-linear (quadratic) regression curve fitting is used, a minimum of 6 calibration points is required (SW8000C). A quadratic regression should not be used to compensate for detector saturation.

8.4.8 The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a “weighting” factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration (1/x) or to the inverse of the square of the concentration (1/x2). If regression criteria cannot be met, system repair or maintenance may be necessary and a new initial calibration must be performed.

The type of curve fit applied should be chosen to best represent the data.

8.5 INITIAL CALIBRATION VERIFICATION (ICV)

A second source (ICV) standard is analyzed immediately after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time. The acceptance criteria for the ICV are identical to those of the CCV (described below). Refer to the QC Table for corrective action should the ICV analysis fail.

8.6 SAMPLE PREPARATION / ANALYSIS

8.6.1 A system blank of air is injected to demonstrate that background is low enough to support analytical goals.

8.6.2 Each VOA vial contains 42.5mL of volume. A 4.0mL headspace was created for the initial MDL study, calibrations and sample analyses, and should therefore be used unless further study is performed to support changing the headspace volume.

8.6.3 Record the room temperature and barometric pressure in the sequence log. The value for barometric pressure may be taken from Colorado State University’s weather observation station. These data show that a value of 840mbar may be used for Fort Collins’ barometric pressure with minimal error.

8.6.4 The sample is equilibrated by vortex mixing at approximately 3000 rpm for at least 2 minutes, or the vials can be tumble at 30 rpm for 60 minutes. Then a 300µL aliquot of headspace is then injected into the GC.

8.6.5 Dilutions. If less than 10% of the original sample headspace was used in a sample analysis, a smaller aliquot, from the same headspace, may be used for gas-phase dilution. The injection size is kept constant. Otherwise, a new sample is prepared at an appropriate dilution. For example, if the head space is 4mL from a 40mL VOA vial and the sample injection is 300µL (7.5% of the headspace), a smaller aliquot of headspace can be used for dilution. The dilution to be performed is chosen to keep the response in the upper half of the calibration curve.

Example Vapor-Phase Dilution: 50µL to 42.5mL (42,500 µL) = 850x

8.7 QC SAMPLES

8.7.1 The following types of QC samples are prepared with each extraction batch of ≤20samples (see QC table for frequency, acceptance criteria and corrective actions):

- Method Blank (MB): No sample added; all reagents and Steps are equivalent to field sample.
- LCS (CCV)/LCSD: Reagent water with equivalent headspace to standard after addition of gas-phase standard. Note that for this procedure, the LCS is equivalent to a CCV (referenced by the method as a CCS). The LCSD is a Duplicate of the LCS.
- MS/MSD: Field sample (and field sample duplicate) fortified with midpoint analyte spike. MSD not required unless specified in the Program Specification or Nickname.
- Duplicate: a field sample duplicate (Dup).

8.7.2 To prepare QC samples, 40mL VOA vials are filled with reagent water (SOP 511) with zero headspace.

8.7.3 Create a 4.0mL headspace

8.7.4 Vials thusly prepared can be method blanks, or can be spiked to create working standards. Note that working standards are equivalent to LCSs for this procedure.

8.8 CONTINUING CALIBRATION VERIFICATION (CCV)

The CCV is used to confirm system response throughout an analytical sequence. The concentration of the CCV is at or around the midpoint of the initial calibration. Acquire a CCV at the start of each analytical sequence, after each twenty injections (or less), and at the end of each sequence. ALS commonly analyses 10 samples between CCVs to reduce the amount of repeat injections, should they be required. QC samples are counted as part of the number of injections, instrument blanks are not.

The percent difference (%D, drift) must be calculated for each CCV (see equation below):

$$\%D = \left[\frac{(\text{calculated concentration}) - (\text{expected concentration})}{\text{expected concentration}}\right](100)$$

Calibration is verified when all compounds are within 20%D. Individual compounds that exceeded 20% are noted in the data package narrative. If any CCV does not meet acceptance criteria, analyses should be halted and corrective action taken. Refer to the QC Table for corrective action in the event of CCV analysis failure.

8.9 RETENTION TIME WINDOWS

For GC methods utilizing external standard quantitation, retention times are used for analyte identification. Retention Time Windows (RTWs) are established each time a new column is installed and are used to compensate for minor retention time shifts. It is important to establish valid retention RTWs. If too tight, false negatives may result. If too loose, false positives may occur. Determine RTWs by analyzing replicates (typically three injections), of a mid-level standard containing all analytes, non-consecutively, over a 72-hour period (this approach captures system variation). Calculate the standard deviation of absolute retention time for each analyte for the set of analyses used in the RTW study. Define each analytes' RTW as the mean retention time $\pm 3\sigma$, such that the Upper Limit = $+ 3\sigma$ and the Lower Limit = -3σ.

8.10 SAMPLE IDENTIFICATION, CALCULATIONS, REPORTING

8.10.1 Dual column confirmation is not required because interferences are not observed due to strong separation/greater retention of interferences on the chromatographic column.

8.10.2 The following equation is used to quantify sample concentration when CF (or mean CF) is employed:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(DF)}{(\text{mean CF})(V_s)}$$

where:

A_x = analyte response (area units)

DF = dilution factor (if applicable); if no dilution was made, DF = 1 (dimensionless)

CF or mean CF = standard response (area units/concentration)

Vs = volume of sample analyzed (L)

8.10.3 Where linear regression is employed, quantitation of sample concentration is based on the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)\,(V_t)(DF)}{m}$$

where:

x = concentration of the analyte (µg/L)

y = analyte instrument response (area units)

b = calculated intercept (area units)

m = calculated slope of the line (area/conc. in µg/L)

V_t = total volume of concentrated extract (L)

DF = Dilution Factor (if applicable);
if no dilution, then DF = 1

9. QUALITY CONTROL

Quality Control Samples are defined in QAM section 14.9.

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specifications for additional or alternative requirements.

9.2 BLANKS

Method Blanks (MBs) are aliquots of matrix (i.e., water) that have been prepared and analyzed in the same manner as the associated field samples. MBs are analyzed to demonstrate that the system overall is under control. Concentrations of target analytes, if any, must be less than the reporting limit (RL), or as otherwise prescribed in the LIMS program specification.

9.3 LABORATORY CONTROL SAMPLE

The LCS is analyzed to measure the accuracy of the analytical system. An LCS is similar to a matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}}\right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample and/or matrix spike sample is performed in duplicate (LCSD, MSD). The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$RPD = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})}\right)(100)$$

9.5 MATRIX SPIKE

The matrix spike is analyzed to measure matrix effects on analyte recovery. To accomplish this, a measured amount of field sample is spiked with a known amount of analyte and % Recovery is calculated as above for the LCS. See the QC Table for evaluation criteria.

$$\%R = \frac{\text{(MS Sample result-Sample result)}}{\text{Spike added}} \times 100$$

9.6 METHOD DETECTION LIMIT STUDY
A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven (7) replicates for each target analyte at a concentration level near to the capabilities of the method. The MDL study is performed as needed, at minimum, annually, following the guidance of SOP 329.

9.7 CORRECTIVE ACTION AND CONTINGENCIES
Any method specific corrective actions as specified in the reference method can be referenced. At a minimum include the following:

- Corrective action specific to the instrumentation

- ALS SOP 928 "Non-Conformance and Corrective Action Procedures

10. DEVIATIONS FROM METHOD

10.1 Method RSK175: Specifies that the instrument blank (helium, nitrogen, or air) acceptance is less than the reporting limit (RL).

10.2 EPA Region 1, Analysis of Dissolved Methane, Ethane, and Ethene in Groundwater by a Standard Gas Chromatographic Technique: Butyl rubber VOA vial septa are specified in the EPA Region 1 method. Teflon-faced silicone septa are commonly employed by ALS for volatile analytes. Adaptation of butyl rubber septa may be validated and adapted for this assay. ALS has tested septa supplied to clients.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS
All Safety and Hazards are managed in accordance with the current facility plans:
- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL
All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 Felisa Hudson. RSKSOP-175. Revision No.2, May 2004.

12.2 Don H. Kampbell and Steve A. Vandegrift. "Analysis of Dissolved Methane, Ethane, and Ethene in Groundwater by a Standard Gas Chromatographic Technique". EPA, Ada, OK. Journal of Chromatography, Vol. 36, May 1998.

12.3 "Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene", Methane, Ethane, Ethene Analysis Guidance, Revision 1. US EPA - REGION 1, New England, NATATTEN.WPD. 11 Technology Dr. North. Chelmsford, MA 01863. February 21, 2002.

Analytical Method: RSK175	**Parameter:** Methane, Ethene, Ethane		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	QC Check
Initial Calibration; minimum 5-point; all analytes	As needed (i.e., when the daily calibration does not meet criteria)	Calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r^2) ≥0.990 (or r = ≥0.995) or calculate quadratic regression (minimum of six points required); use for quantitation if COD ≥0.990 ≤20%D each point	Evaluate/correct instrument malfunction and reanalyze ICAL to obtain acceptable curve
Independent Calibration Verification (ICV); all analytes	After each new initial calibration	≤20%D of each compound Note: Second lot is acceptable if second source is unavailable.	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.
Continuing Calibration Verification (CCV); analyzed at approximately midpoint concentration level of the calibration curve	Run at start of sequence if ICAL not performed; brackets each set of 10 (or max. of 20) field sample analyses	≤20%D for each analyte or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g. change septum, rinse or change liner; prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. Samples analyzed before and after a failed CCV must be reanalyzed.
Retention Time Window (RTW); based on minimum of 3 non-consecutive injections throughout at least a 72-hour period to be representative of variation	Update whenever a new column is installed or target analytes are misidentified in a standard, LCS or MS	Column and compound specific Window is ±3x the standard deviation of the 3-injection average for the respective column Note that the ICV and CCV analyses are also used to monitor RT drift	Wider windows can be used to screen for compounds; if zero, substitute window of close eluting similar compound. Experience of analyst weighs heavily in interpretation of chromatograms (refer also to RT Shift).
Retention Time Shift; RT of analytes in CCV are evaluated against the midpoint of the ICAL	Each CCV; RT of analytes evaluated against the ICAL	Column and compound specific, must support consistent identification of analytes in know samples.	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate Evaluate data based on comparison with other standards run during sequence, consider RTs for the surrogates and spiked compounds analyzed before and after the sample in question: - adjust the RTW to correct the shift in compound location - if no peaks are found in the adjusted window, report the compound as a non-detect

Analytical Method: RSK175	**Parameter:** Methane, Ethene, Ethane		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	QC Check
			- if peaks are present, use the confirmation column to verify identification
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	If not less than acceptable limit, correct contamination and re-analyze associated samples if possible. Note: Due to the ubiquitous nature of methane, method blanks may occasionally have concentrations >RL. Such incidents are acceptable if concentrations of methane in associated samples are either ≥5x the concentration in the method blank OR <RL for methane. If the above conditions are met, then no Non-Conformance Report (NCR) needs to be generated. If the above conditions are not met, then consult the Project Manager for guidance regarding an NCR and corrective action.
Blank Spike (BS); Laboratory Control Sample (LCS); Since an LCS is physically equivalent to a CCV, a CCV may be designated as "CCS" and used as an LCS.	1 per preparation batch of ≤20 samples of like matrix	80-120% Recovery or as specified in individual nicknames; recoveries for spiked compounds must be within these limits or other limits as specified in the LIMS program specification	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed) - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration
Matrix Spike (MS) Matrix Spike Duplicate (MSD) or sample Duplicate	1 per preparation batch of ≤20 samples of like matrix	70-130% Recovery or per applicable Nickname/Program Specification; recoveries for spiked compounds should be within advisory limits The relative percent difference (RPD) between duplicate analysis (sample/sample duplicate or MS/MSD) should	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the

Analytical Method: RSK175	**Parameter:** Methane, Ethene, Ethane		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	QC Check
		be ≤ 20 (or per Nickname/Program Specification requirement)	MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers. Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed)

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	24.0
EFFECTIVE DATE:	06/03/2017

ALS Environmental

STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOP ID: VOA-TO15 Rev. Number: 24.0 Effective Date: 06/03/2017

Approved By: Wida Ang Date: 5/25/17
Team Leader (VOA GC/MS) - Wida Ang

Approved By: Date: 5/26/17
Technical Services Manager - Chris Parnell

Approved By: Date: 5/31/17
QA Manager - Chaney Humphrey

Approved By: Date: 5/31/17
Laboratory Director - Kelly Horiuchi

Archival Date: ________ Doc Control ID#: UNCONTROLLED Editor: ________

SOP CHANGE FORM

SOP Title: Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS)
SOP Code: VOA-TO15
SOP Revision No.: 24.0
SOP Date: 06/03/2017
SOP Section(s) Affected by Change: Tables 2, 2A, 3, 3A, 4, 4A

Description of Change: Add the following to Tables 2, 2A, 3, 3A, 4, 4A:

Table 2 – Volatile Organic Compounds, EPA Compendium Method TO-15 (Scan)

Compound	CAS Number	Molecular Weight	Primary Ion	Secondary Ion	MRL (ug/m3)	MDL (ug/m3)	IS
Bromobenzene	108-86-1	157.01	77	156, 158	0.50	0.25	IS3

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Compound	Primary Ion	Secondary Ion	MRL (ug/m3)	MDL (ug/m3)	IS
Bromobenzene	77	156, 158	0.10	0.0042	IS3

Table 3 – Standard Concentrations (Scan) (Primary Sources)

Compound	0.4ng	1.0ng	2.5ng	5.0ng	25ng	50ng	100ng
Bromobenzene	0.424	1.06	2.65	5.30	26.5	53.0	106

Table 3A – Standard Concentrations (SIM) (Primary Sources)

Compound	20pg	50pg	100pg	200pg	500pg	2000pg	5000pg	10000pg
Bromobenzene	21.2	53.0	106	212	530	2120	5300	10600

Table 4 – Standard Concentrations (SCAN) (Secondary Sources)

Compound	25ng
Bromobenzene	26.50

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Table 4A - ICV/LCS Standard Concentrations (SIM) (Secondary Sources)

Compound	500pg
Bromobenzene	530.0

Reason(s) for Change(s): Add bromobenzene to list of compounds.

Change(s) Submitted by: By Chaney Humphrey at 2:38 pm, Oct 13, 2017	Date: 10/13/17

Approvals:

QA Manager Signature: By Chaney Humphrey at 2:38 pm, Oct 13, 2017	Date: 10/13/17
Supervisor/Manager Signature: Wida Ang Wida Ang	Date: 10/13/17

Change(s) Effective Date: October 13, 2017

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters or glass bottles. This method typically applies to ambient concentrations of VOCs 0.50ug/m3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity. Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

HAZARD ASSESSMENT

Job Task 1	**Hazards**	**Preventative Measures**
Standard and Sample Preparation. Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure. Be familiar with proper safety precautions.
Job Task 2	**Hazards**	**Preventative Measures**
Working with Liquid Nitrogen: Turning valves and handling tubing and fittings that have been in contact with cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task 3	**Hazards**	**Preventative Measures**
Working with Pressurized Gases: Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task 4	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	Proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include.

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. See Attachment 5 for trap packing instructions. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of three hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.5). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

5.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

5.6 Computer Troubleshooting

Computer care and troubleshooting is conducted by the IT department. Refer to Section 9.6 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

6) Interferences

6.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 16.7 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

6.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

6.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning*. The use of high purity water, reagents, and solvents helps to minimize these problems.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

7.4.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.

2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.1.4.2 (LOQ) and 12.14.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.4.2 Annual Demonstration Each analyst must perform a demonstration of capability initially and annually. For the initial demonstration analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

7.4.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.4.1 and 7.4.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L), a glass sampling bottle (Bottle Vac, Entech Inntruments) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP for Flow Controllers and Critical Orifices*) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

8.2 There are no special preservation requirements for either canisters, Bottle Vacs or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters and bottles should be stored on the appropriate shelves until they are to be analyzed.

8.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. “The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days.”

9) Equipment and Supplies

9.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

9.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series
Agilent 7890B Series

9.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Markes Autosampler:	UNITY 2/CIA Advantage
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

9.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every 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 Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*
Agilent 5977A

9.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

9.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

  OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2011 version or newer) or equivalent.

9.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

9.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller
- Critical Orifices (Laboratory manufactured)

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent
- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

10) Standards and Reagents

10.1 Reagents and Equipment

10.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)

10.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

10.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

10.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan User's Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500μg/m^3 each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 10.2.2.2.1 or as stated in Section 10.2.1.3.

10.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0μg/ml. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60°C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0μg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A	Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C	Desired concentration of SDB (µg/mL)
V	Actual volume of the SDB (mL)
D	Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0 \frac{\mu g}{mL}\right) 2010\, mL}{1934.4 \frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 10.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV	Pressurized canister volume (L)
PDF	Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f	Final Canister Pressure
P_i	Initial Canister Pressure
V	Volume of canister at 1atm
P_{atm}	Atmospheric Pressure = 14.7psig

Example:

$$\frac{14.7+83.3}{14.7+0}(6L) = 40\text{L}$$

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$\text{A}=\frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \qquad \text{(Equation 3)}$$

Where:

F	Desired concentration of working standard (ng/L)
V	Pressurized Volume of Canister (L)
C	Concentration of prepared SDB (µg/mL)
A	Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$\text{A} = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4\text{mL}$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ).

The internal standard (IS) cylinder comes from the vendor with a one year expiration date. These compounds should be stable in the high-pressure cylinder for five years or longer so the laboratory will extend the expiration date to two years from the date of preparation. The working standards are Summa canisters filled directly from the main cylinder and are given a two month expiration period. The method utilized relative response factors for target analyte quantitation so the IS concentrations are factored out since they appear in the numerator and denominator of the final calculation.

A quantitation report with chromatogram of a TO-15 blank run will be printed as soon as a new IS cylinder is put into use and again after one year. The latter will be checked for any unexpected peaks to look for possible degradation of the IS compounds in the cylinder. These shall be kept on file with the original certificate of analysis.

10.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration using Equation 2 in Section 10.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

10.2.2 <u>Initial Calibration (ICAL) Standard</u> Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 1ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L, 5.0ng/L, and 200ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

10.2.2.1<u>Working standards</u> are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the three working standards prepared in Summa canisters should be 200ng/L, 20ng/L and 1ng/L (depending on the dynamic range of the initial calibration include 1ng/L if a 0.08ng and 0.4ng on column standard is desired <u>or</u> this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.2ng/L, 4.0ng/L, and 200ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)
Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

<u>Step1</u>: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final

dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min
1ng/L	50ml/min; 20ml/min (See Note 1 below)
0.2ng/L	10ml/min; 20ml/min (See Note 1 below)

Note 1: For the 1ng/L and 0.2ng/L standards (or any standard requiring more than a 400X dilution of the stock), a slightly different procedure is performed. In order to prepare these standards, a double dilution must be performed which involves taking the primary dilution flow and making a secondary dilution of that using the diluent gas. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.2ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.2ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.2ng/L = 5,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50
Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

10.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 10.2.2.2.1 – 10.2.2.2.2) as mentioned in Section 10.2.1.

10.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:

S Actual spike amount (µL)
A Desired amount for each compound (mg)
D Density (mg/µL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(\text{Acrolein}) = \frac{25mg}{\left(0.840\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (µg/µL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (µL)

Example:

$$C = \frac{25mg}{631.8\mu L}\left(1000\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

10.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 10.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

Where:

S Spike amount required in order to obtain the desired concentration (µL)
C_1 Desired concentration of SDB (µg/mL)
C_2 Concentration of cocktail (µg/µL)
V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

10.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds)* As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\,\frac{ng}{\mu l}\right)}$$

S	Spike amount required in order to obtain the desired concentration (µl)
C	Desired concentration (ng/L)
V	Volume of the Tedlar Bag (1L)
M	Molecular Weight of the compound
24.46	Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

$$S = \frac{100{,}000\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\frac{ng}{\mu l}\right)} = 28.44\mu l$$

10.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 10.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 10.2.2, using the certified second-source standard cylinder.

10.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 10.2.2.

10.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

10.3 Storage and Expiration Dates

- All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.
- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards. Expiration dates of the cylinders must be entered into the yearly wall calendar located next to the cylinders. Analysts must verify that the assigned expiration dates of prepared standard canisters do not exceed the parent standard expiration date.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an

atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).

- Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

  When an individual compound point is dropped from an ICAL both the response and concentration fields in the compound database of the method must be cleared. This ensures the average ICAL RRF calculates correctly when executing the CCV check routine.

- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
- It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.4ng point).

11.1.1 <u>Calibration Points</u> Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.4ng-100ng on column; however, 0.08ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 50,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

<u>Note</u>: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

<u>SCAN</u>

1. Determine if the lower end of the calibration range is to be 0.08ng or 0.4ng on column. If the low end is 0.08ng, then the 1ng/L standard must be utilized.
2. Determine if the 1ng/L or 20ng/L standard canister is to be used for the 0.4ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.1.2 <u>Recalibration</u> Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.
7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 12.3.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.4. Use the standard prepared in accordance with Section 10.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 15.2 for the required calculations and Section 16.4 for the acceptance criteria.

11.1.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ is verified by analyzing an LOQ verification QC sample containing the analyte at 1-2 times the claimed LOQ.
2. The LOQ for each analyte must be > the analyte's LOD.
3. The verification is acceptable if:
   a. The S/N ratio is at least 3:1 for each analyte.
   b. All ion abundances are acceptable per the requirements in this document.
   c. The % recovery for each analyte is within the laboratory generated control limits or 70-130% recovery for the annual Navy LOQ verification.
4. Using from 2 to 4 LOQ verification points, calculate the ongoing %RSD to demonstrate precision at the LOQ.
5. If the LOQ verification check fails, determine and document the cause. Additional LOQ verification checks must be performed at a higher level to set a higher LOQ.
6. Turn in all LOQ verification data (quantitation reports and software reports/checks) to QA regardless of pass or fail.
7. Verify the LOQ on each instrument quarterly. Navy accreditation requires an annual LOQ verification.

11.1.5 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.1.6 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

11.2 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 15.4 for the required calculations.

12) Sample Preparation/Analysis

12.1 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 12.9 for additional information). The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 15.7.

12.2 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 10.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 12.9.1.

$$I = \frac{C}{H}$$

Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100\text{mL maximum injection volume}$$

12.3 Analytical Sequence and Data System Setup

12.3.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.4.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.4.2.

12.3.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6]

MB[7]
LCS[3]
MRL Check Standard[8]
Sample(s) – 1-19
Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 12.15.

Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

12.4 Conditions

12.4.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point: 35°
Sample Volume: up to 1L
Dry Purge: 300mL
Sampling Rate: 100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.: 200°C to 230°C
Desorb Flow Rate: 8-10mL/min He, measured at refocuser split vent
Desorb Time: 3.0 minutes

Refocusing Trap

Temperature: -180°C
Injection Temp.: 160°C

Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C
Initial Bakeout:	3 hours or until clean blank is obtained
After each run:	5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: ~20°C Initial Hold Temperature: 3 minutes Ramp Rate: 5°C/min to 80°C 2nd Ramp: 10°C/min to 160°C 3rd Ramp: 20°C/min to 240°C for 5 min hold
Detector B (MSD *Interface)*	260°C
Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

12.5 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

12.6 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 15.3 for the required calculations.

DoD QSM 5.1 Requirement: A CCV standard must be analyzed daily before sample analysis; after every 24 hours of analysis time; and at the end of the analytical batch run.

12.7 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected. For all method blanks the unique laboratory barcode for the canister must be included in the sample analysis identification.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 16.7 and 16.8) and other requirements (refer to Section 16.12 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

12.7.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus performance of the relative accuracy certification procedure described in section 8.4.4 is not possible.

12.8 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Tedlar bags can be connected using soft silicone tubing or a 3/16" fitting with a reusable ferrule.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 12.13).

Note 1: *The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).*

Note 2: *Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.*

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 12.4.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 12.9.2 for identification criteria.

SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.9.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 15ml to 1L. If a volume smaller than 15ml is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 12.4.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:

- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:

- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.
- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

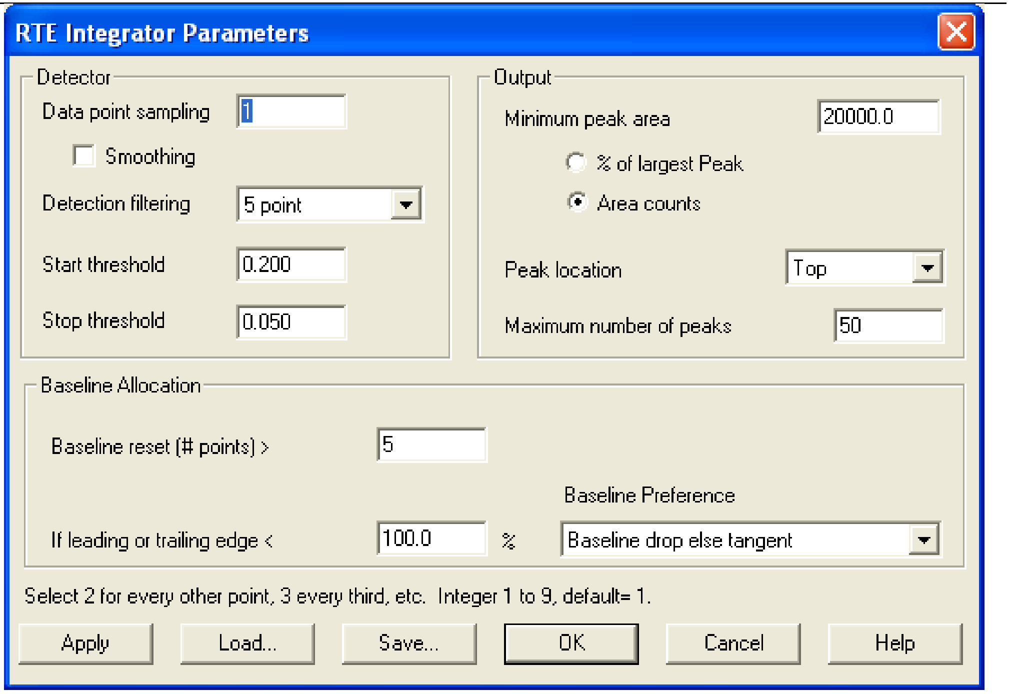
12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

<u>Procedure for Reporting Tentatively Identified Compounds (TICs) for samples and associated Method Blanks</u>

1. Load the datafile in the main Enviroquant window.
2. Load the TIC integration parameters (LSCINT.p). Typical setpoints are as shown below.



Data point sampling: 1
☐ Smoothing
Detection filtering: 5 point
Start threshold: 0.200
Stop threshold: 0.050

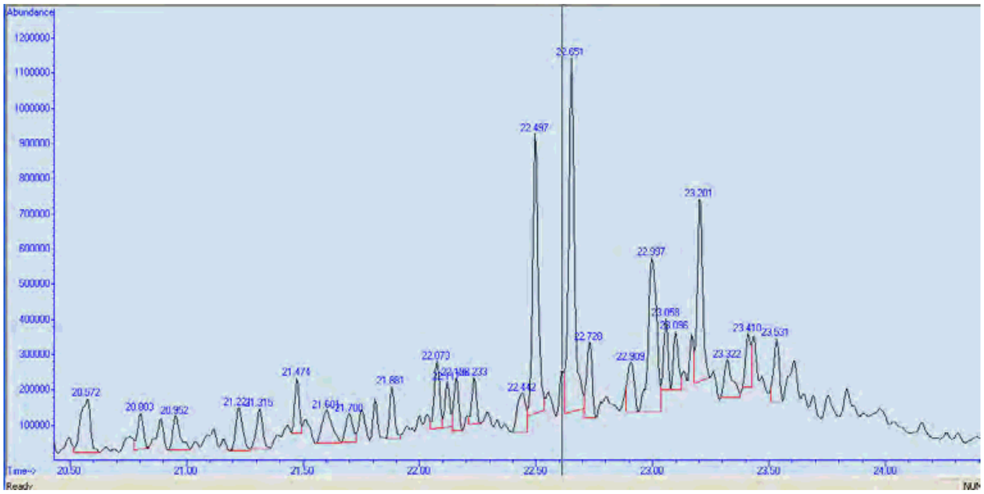
Minimum peak area: 20000.0
○ % of largest Peak
● Area counts
Peak location: Top
Maximum number of peaks: 50

Baseline reset (# points) > 5
If leading or trailing edge < 100.0 %
Baseline Preference: Baseline drop else tangent

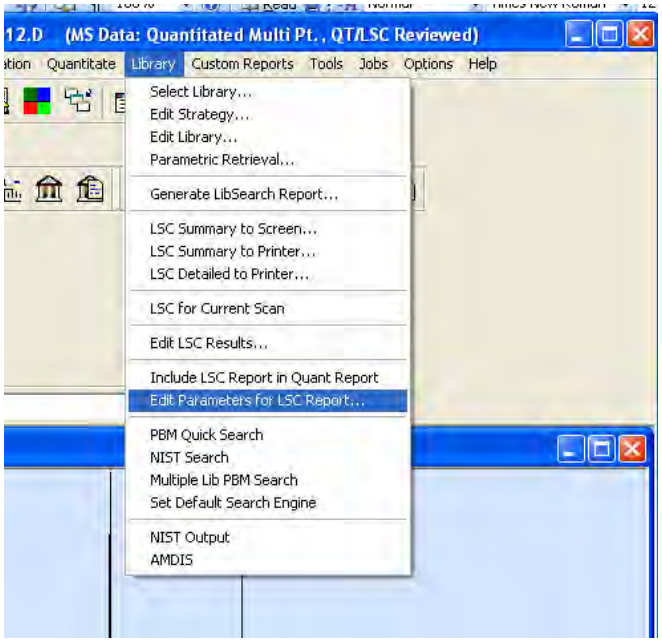
Select 2 for every other point, 3 every third, etc. Integer 1 to 9, default= 1.

3. Integrate the chromatogram and inspect the peak integrations. Adjust the parameters as needed to achieve integration that will:

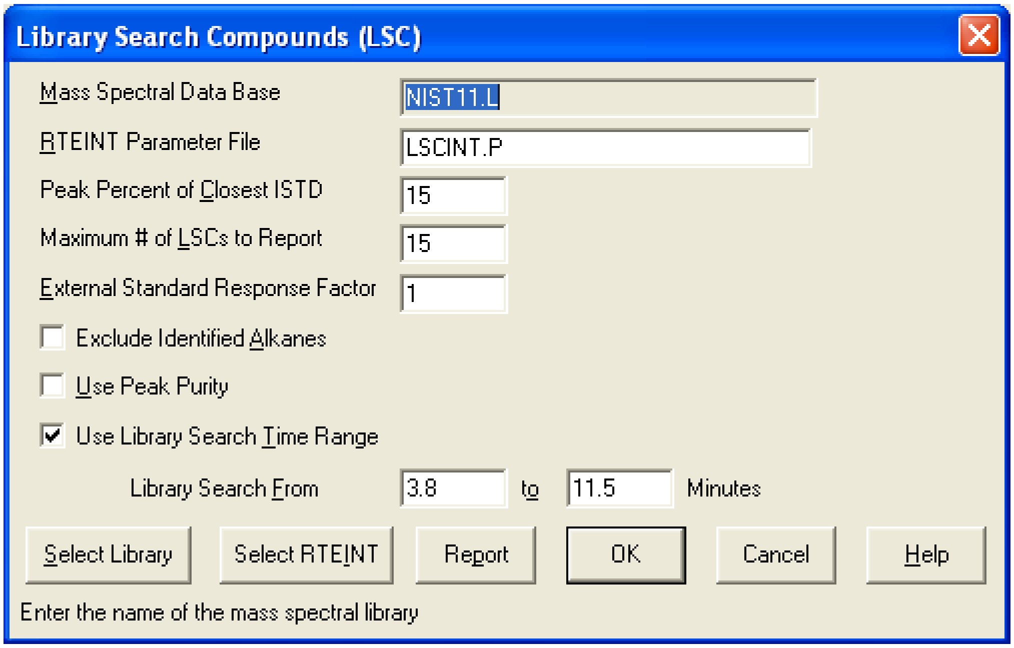
   - Resolve closely-eluting peaks that only have a small valley separating them.
   - Not include excess area below the peak in a complex matrix with an elevated baseline.
   - Include peak tailing when necessary.
   - Yield a sufficient number of peaks that will ensure that the internal standards are included.



4. Edit the parameters to be used in generation the library search report:



Select the most current mass spectral library database available, the correct integration parameters file, the area threshold (as a percent of IS area), number of peaks to report, and a time range of the chromatogram to search (set to start after the CO2 peak).



Mass Spectral Data Base: NIST11.L
RTEINT Parameter File: LSCINT.P
Peak Percent of Closest ISTD: 15
Maximum # of LSCs to Report: 15
External Standard Response Factor: 1
☐ Exclude Identified Alkanes
☐ Use Peak Purity
☑ Use Library Search Time Range
Library Search From 3.8 to 11.5 Minutes

Enter the name of the mass spectral library

5. Run the LSC routine from the Library menu. You may choose ‘LSC Summary to Screen’ (Calculate/Generate Report) to get a quick view of the results and then proceed if they seem acceptable. Set the default printer to ‘Adobe PDF’ and then choose ‘LSC Detailed to Printer’.
6. Open the pdf file and inspect the LSC summary (last page). Check the internal standard areas and confirm that they are correct. If any IS area is biased high due to a coeluting peak use the ‘Edit LSC Results’ routine to switch all associated TICs to use a different IS. If all three IS peaks have coelutions substitute the areas from the daily method blank in the calculations.
7. Use the LSC Summary as a guide and inspect the chromatogram in the data analysis window. Integrate the chromatogram from the Integrate menu and look for peaks that may have been missed by the LSC routine. Possible reasons for missed peaks are excessive tailing (organic acids), RT close to a target compound, coeluting peaks with no valley between them. These will need to be added manually.
8. Use the DOSCAN routine from the Tools menu to search individual missed peaks one by one. This will add them to the LSC list.
9. Go back into the Edit LSC Results routine and make any necessary changes to compound names and/or the internal standard used for quantitation.
10. Run the macro “QT ‘0,0,C’ by clicking the Custom Tool 1 button. This will update the LSC list to the quant.csv file.
11. Run the LSC Detailed to Printer routine from the Library menu (Generate Report *only*). This will print the file to pdf.
12. Excel Reporting
    1. In Excel, open the TIC reporting template (I:\A-GCMS\TICS\System\StarLIMS_TICQ).
    2. Enter the service request number and click ok.
    3. Click the Get Samples button. Select the samples to be reported. Delete any samples that are not to be reported (right click/delete row).
    4. Click the Update PEF button.
    5. Click the Get TICs from CSV button. Enter the date analyzed and select the instrument ID.
    6. Click the Apply to all Samples button. Change the date for any sample that was analyzed on a different date.
    7. Click the Apply Instrument to all Samples button.
    8. Enter file number in column E (i.e. enter 07 for file 12301507.d).
    9. Click the Copy Data button. This copies the TIC info to the report sheets.

12.10 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

12.11 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

12.13.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

12.13.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

12.14 Detection Limits and Limits of Detection

The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

12.14.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Following the MDL study perform a Limit of Detection (LOD) verification on all instruments (performing this method). Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.

5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

12.15 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.1.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment 4 for Minnesota specified MRL check standard criteria.

12.16 Method Modifications

Method modifications are not allowed under TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.2 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration.

This includes all samples, continuing calibrations and QC samples.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 15.10 for additional information and specific items, which require this clarification.

15.2 Initial Calibration

Tabulate each of the following:

15.2.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$\text{RRF} = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: *The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.*

15.2.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$

where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

15.2.3 Equation Number 3 - **Standard Deviation, SD:**

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$$ where:

RRF_i	are the individual RRFs from each concentration level in the initial calibration curve.
$\overline{RRF}$	Average (or Mean) RRF of all concentration levels in the initial calibration curve.
N	total number of calibration concentration levels

15.2.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

$$\%RSD = \frac{SD}{\overline{RRF}}(100)$$ where:

SD	Standard Deviation calculated in equation number 3
$\overline{RRF}$	Average or Mean RRF

15.2.5 Equation Number 5 - **Relative Retention Time (RRT):**

$$RRT = \frac{RT_C}{RT_{is}}$$ where:

RT_C	Retention time of the target compound, seconds.
RT_{is}	Retention time of the internal standard, seconds.

15.2.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$ where:

$\overline{RRT}$	Mean relative retention time (seconds) for the target compound for all initial calibration levels.
RRT_i	Relative retention time for the target compound in level i.
n	Number of calibration levels

15.2.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$ where:

Y_i	Area response for the primary quantitation ion for the internal standard for each initial calibration standard.
n	number of calibration concentration levels

15.2.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$ where:

$\overline{RT}$ Mean retention time, seconds

RT_i Retention time for the internal standard for each initial calibration standard, seconds.

n number of initial calibration levels

15.3 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1.

15.3.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$ where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.

$\overline{RRF}$ is the mean RRF from the current calibration curve.

15.4 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

15.4.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

15.5 Duplicate Analysis

15.5.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\overline{x}}(100)$$ where:

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

15.6 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7.
- Calculate the mean of the retention times for each internal standard using equation number 8.

15.7 Pressure Dilution Factor (PDF)

15.7.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$\text{PDF} = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.

P_f is the final sample canister pressure, in psig.

P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

15.8 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 12.9.

15.8.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x.*

A_x is the area response of the analyte's quantitation ion.

A_{is} is the area response of the corresponding internal standard's quantitation ion.

ng_{is} is the internal standard amount, in nanograms.

$\overline{RRF}$ is the average or mean RRFs

15.8.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.

PDF is the sample canister pressure dilution factor.

15.8.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW} x24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46} xMW$$ where:

MW is the molecular weight (Table 2) of the analyte, in g/mole.

24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\System\HE Pressurization Template.

15.9 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the Daily QC review checklist (Attachment 3). The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The Sample Review checklist (Attachment 3) is used to document sample review per service request and once completed, initialed and dated must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.6.

15.10 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, may be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form may be used as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. Alternatively, information may be included on the Daily QC and Sample Review Checklists (Attachment 3). All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAP\TNI Requirements

The following items do not comply with TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAP requirements.
- In addition, a report that contains a compound not included on the NELAP certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

Method modifications are also not allowed under TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).

  DoD QSM 5.1/Navy Requirement: The two exceptions of %RSD up to 40%, allowed by the method, are not allowed.

- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.6.2.1 below.

DOD Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (The two consecutive CCVs must be analyzed within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.6.2.1 Method Reporting Limit Check Standard

If a per batch MRL check standard is analyzed due to a failing CCV or client requirement and is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly. Reporting data with these conditions must be acceptable per project and client requirements otherwise corrective action must be initiated and samples reanalyzed.

Refer to Section 11.1.4.2 for annual (NELAP and Navy) and quarterly (DoD) LOQ verification requirements.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered “clean” for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

DoD QSM 5.1 Requirement Each canister must be individually certified. A canister is considered clean if no reported analytes are detected at >1/2 the LOQ.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:
  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit or is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds

the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or

3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior;

therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.
- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). A percent difference +/-50% is recommended but program and client specific requirements must be followed if applicable.

16.15 Sample Holding Time Expired

The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).
5. Data associated with a biased low CCV may be fully useable if the results reported exceed a maximum regulatory limit/decision level.

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 2011 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM.

Proficiency testing samples including all accredited compounds are not available. Therefore, in addition to third party PT samples, intra laboratory comparisons must be performed biannually to meet the DoD QSM proficiency testing requirements. Eight QC analyses from various analysts and instruments shall be compiled and statistical validity evaluated using a Z-score.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
24.0	06/03/17	C. Humphrey	5.2 – Included reference to Attachment 5; changed bake time to three hours
		C. Humphrey	7.4 – Removed DoD QSM version number
		C. Humphrey	7.4.2 – Minor wording revision
		C. Parnell	9.6 – Updated NIST Library to 2011
		C. Parnell	12.4.1 – Added information to Desorb Flow Rate; changed bake time to 3 hours under Adsorbent Trap Reconditioning Conditions
		C. Humphrey	12.6 – Added DoD QSM 5.1 requirement
		C. Humphrey	12.14 – Revised to align with current procedure and SOP CE-QA011
		C. Humphrey	12.14.1 – Revised to align with current procedure and SOP CE-QA011
		C. Humphrey	15.8.4 – Updated file path
		C. Humphrey	15.10.1 – Revised to align with current procedure
		C. Humphrey	16.4.1 – Added DoD QSM 5.1/Navy requirement
		C. Humphrey	16.7.1 – Added DoD QSM 5.1 requirement
		C. Humphrey	16.8.1 – #1 changed “and” to “or” to align with DoD QSM version 5.1
		C. Humphrey	16.9.1 – Removed DoD QSM version number
		C. Parnell	19.4 – Updated NIST Library to 2011
		C. Humphrey	19.6 – Revised section
		C. Humphrey	21.7 – Updated reference
		C. Humphrey	21.8 – Updated reference
		C. Humphrey	22.2 – Included Attachment 5
		C. Humphrey	Updated Tables 2A, 3, 3A, 4, 4A
		C. Humphrey	Attachment 2 – Added #15 and renumbered; #17 revised wording
		C. Humphrey	Attachment 3 – Added #5 and renumbered; Added #12
		C. Parnell	Attachment 5 - New

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 2009 TNI Standards

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 DoD/DoE Quality Systems Manual Version 5.0, 2013; and Version 5.1, 2017.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, October 1, 2016.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

22.2 Attachments

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m^3)	MDL[3] (µg/m^3)	IS[4]
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m^3)	MDL[3] (µg/m^3)	IS[4]
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (μg/m^3)	MDL[3] (μg/m^3)	IS[4]
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m^3)	MDL[3] (µg/m^3)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Compound	Primary Ion[1]	Secondary Ion[1]	MRL[2] (ug/m3)	MDL[2] (ug/m3)	IS
Dichlorodifluoromethane	85	87	0.050	0.017	IS1
Chloromethane	52	50	0.050	0.019	IS1
Vinyl Chloride	62	64	0.025	0.0076	IS1
1,3-Butadiene	54	39	0.050	0.014	IS1
Bromomethane	94	96	0.025	0.0093	IS1
Chloroethane	64	66	0.025	0.0085	IS1
Acrolein	56	55	0.20	0.039	IS1
Acetone	58	43	2.5	0.056	IS1
Freon 11	101	103	0.050	0.015	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0086	IS1
Methylene Chloride	84	49	0.10	0.013	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0089	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0073	IS1
1,1-Dichloroethane	63	65	0.025	0.0061	IS1
Methyl tert-Butyl Ether	73	57	0.025	0.0093	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0092	IS1
Chloroform	83	85	0.10	0.018	IS1
1,2-Dichloroethane	62	64	0.025	0.0084	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0059	IS1
Benzene	78	77	0.075	0.020	IS1
Carbon Tetrachloride	117	119	0.025	0.012	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0073	IS2
Bromodichloromethane	83	85	0.025	0.0069	IS2
Trichloroethene	130	132	0.025	0.0085	IS2
1,4-Dioxane	88	58	0.10	0.0085	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0062	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0055	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0079	IS2
Toluene	91	92	0.10	0.011	IS2
Dibromochloromethane	129	127	0.025	0.0088	IS3
1,2-Dibromoethane	107	109	0.025	0.0079	IS2
Tetrachloroethene	166	164	0.025	0.0082	IS2
Chlorobenzene	112	114	0.10	0.0092	IS3
Ethylbenzene	91	106	0.10	0.0097	IS3
m-&-p-Xylene	91	106	0.10	0.019	IS3
Styrene	104	103	0.10	0.0074	IS3
o-Xylene	91	106	0.10	0.0089	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0072	IS3
1,3,5-Trimethylbenzene	105	120	0.10	0.0073	IS3
1,2,4-Trimethylbenzene	105	120	0.10	0.0083	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0085	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0081	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0095	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.013	IS3
Naphthalene	128	129	0.10	0.016	IS3
Hexachlorobutadiene	225	227	0.10	0.0092	IS3

NA = Not Available (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Propene	0.08288	0.2072	0.4144	1.036	5.180	25.900	51.80	103.6
Dichlorodifluoromethane (CFC 12)	0.08376	0.2094	0.4188	1.047	5.235	26.175	52.35	104.7
Chloromethane	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
Vinyl Chloride	0.08184	0.2046	0.4092	1.023	5.115	25.575	51.15	102.3
1,3-Butadiene	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Bromomethane	0.07944	0.1986	0.3972	0.993	4.965	24.825	49.65	99.3
Chloroethane	0.08072	0.2018	0.4036	1.009	5.045	25.225	50.45	100.9
Ethanol	0.41656	1.0414	2.0828	5.207	26.035	130.175	260.35	520.7
Acetonitrile	0.08368	0.2092	0.4184	1.046	5.230	26.150	52.30	104.6
Acrolein	0.08328	0.2082	0.4164	1.041	5.205	26.025	52.05	104.1
Acetone	0.42504	1.0626	2.1252	5.313	26.565	132.825	265.65	531.3
Trichlorofluoromethane	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
Isopropyl Alcohol	0.16840	0.4210	0.8420	2.105	10.525	52.625	105.25	210.5
Acrylonitrile	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
1,1-Dichloroethene	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
tert-Butanol	0.16912	0.4228	0.8456	2.114	10.570	52.850	105.70	211.4
Methylene Chloride	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Allyl Chloride	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
Trichlorotrifluoroethane	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
Carbon Disulfide	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
trans-1,2-Dichloroethene	0.08536	0.2134	0.4268	1.067	5.335	26.675	53.35	106.7
1,1-Dichloroethane	0.08160	0.2040	0.4080	1.020	5.100	25.500	51.00	102.0
Methyl tert-Butyl Ether	0.08528	0.2132	0.4264	1.066	5.330	26.650	53.30	106.6
Vinyl Acetate	0.42120	1.0530	2.1060	5.265	26.325	131.625	263.25	526.5
2-Butanone (MEK)	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
cis-1,2-Dichloroethene	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
Diisopropyl Ether	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Ethyl Acetate	0.17032	0.4258	0.8516	2.129	10.645	53.225	106.45	212.9
n-Hexane	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
Chloroform	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
1,2-Dichloroethane-d4 (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Tetrahydrofuran	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Ethyl tert-Butyl Ether	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
1,2-Dichloroethane	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
1,4-Difluorobenzene(IS2)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
1,1,1-Trichloroethane	0.08592	0.2148	0.4296	1.074	5.370	26.850	53.70	107.4
Isopropyl acetate	0.16832	0.4208	0.8416	2.104	10.520	52.600	105.20	210.4
1-Butanol	0.16840	0.4210	0.8420	2.105	10.525	52.625	105.25	210.5

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	**0.08ng**	**0.2ng**	**0.4ng**	**1.0ng**	**5.0ng**	**25ng**	**50ng**	**100ng**
Benzene	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
Carbon Tetrachloride	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
Cyclohexane	0.17040	0.4260	0.8520	2.130	10.650	53.250	106.50	213.0
tert-Amyl Methyl Ether	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,2-Dichloropropane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Bromodichloromethane	0.08528	0.2132	0.4264	1.066	5.330	26.650	53.30	106.6
Trichloroethene	0.08480	0.2120	0.4240	1.060	5.300	26.500	53.00	106.0
1,4-Dioxane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Isooctane	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
Methyl Methacrylate	0.16880	0.4220	0.8440	2.110	10.550	52.750	105.50	211.0
n-Heptane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
cis-1,3-Dichloropropene	0.08928	0.2232	0.4464	1.116	5.580	27.900	55.80	111.6
4-Methyl-2-Pentanone	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
trans-1,3-Dichloropropene	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
1,1,2-Trichloroethane	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Chlorobenzene-d5 (IS3)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Toluene-d8 (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Toluene	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
2-Hexanone	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Dibromochloromethane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
1,2-Dibromoethane	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
n-Butyl Acetate	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
n-Octane	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Tetrachloroethene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Chlorobenzene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Ethylbenzene	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
m- & p-Xylene	0.16984	0.4246	0.8492	2.123	10.615	53.075	106.15	212.3
Bromoform	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
Styrene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
o-Xylene	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
n-Nonane	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,1,2,2-Tetrachloroethane	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
4-Bromofluorobenzene (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Cumene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
alpha-Pinene	0.08352	0.2088	0.4176	1.044	5.220	26.100	52.20	104.4
n-Propylbenzene	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
3-Ethyltoluene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
4-Ethyltoluene	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
1,3,5-Trimethylbenzene	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
alpha-Methylstyrene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
2-Ethyltoluene	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
1,2,4-Trimethylbenzene	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
Benzyl Chloride	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
1,3-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
1,4-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
sec-Butylbenzene	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
p-Isopropyltoluene	0.08216	0.2054	0.4108	1.027	5.135	25.675	51.35	102.7
1,2,3-Trimethylbenzene	0.08216	0.2054	0.4108	1.027	5.135	25.675	51.35	102.7
1,2-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
d-Limonene	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
1,2-Dibromo-3-Chloropropane	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
n-Undecane	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,2,4-Trichlorobenzene	0.08344	0.2086	0.4172	1.043	5.215	26.075	52.15	104.3
Naphthalene	0.08664	0.2166	0.4332	1.083	5.415	27.075	54.15	108.3
n-Dodecane	0.08360	0.2090	0.4180	1.045	5.225	26.125	52.25	104.5
Hexachlorobutadiene	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
Methacrylonitrile	0.08520	0.2130	0.4260	1.065	5.325	26.625	53.25	106.5
Cyclohexanone	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
tert-Butylbenzene	0.08408	0.2102	0.4204	1.051	5.255	26.275	52.55	105.1
n-Butylbenzene	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	5000pg	10,000pg	25,000pg	50,000pg
Freon-12	10.47	20.94	52.35	104.7	523.5	1047	5235	10470	26175	52350
Chloromethane	10.05	20.10	50.25	100.5	502.5	1005	5025	10050	25125	50250
Vinyl Chloride	10.23	20.46	51.15	102.3	511.5	1023	5115	10230	25575	51150
1,3-Butadiene	10.57	21.14	52.85	105.7	528.5	1057	5285	10570	26425	52850
Bromomethane	9.93	19.86	49.65	99.3	496.5	993	4965	9930	24825	49650
Chloroethane	10.09	20.18	50.45	100.9	504.5	1009	5045	10090	25225	50450
Acrolein	10.41	20.82	52.05	104.1	520.5	1041	5205	10410	26025	52050
Acetone	53.13	106.26	265.65	531.3	2656.5	5313	26565	53130	132825	265650
Freon-11	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
1,1-Dichloroethene	10.59	21.18	52.95	105.9	529.5	1059	5295	10590	26475	52950
Methylene Chloride	10.57	21.14	52.85	105.7	528.5	1057	5285	10570	26425	52850
Freon-113	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
trans-1,2-Dichloroethene	10.67	21.34	53.35	106.7	533.5	1067	5335	10670	26675	53350
1,1-Dichloroethane	10.20	20.40	51.00	102.0	510.0	1020	5100	10200	25500	51000
Methyl tert-Butyl Ether	10.66	21.32	53.30	106.6	533.0	1066	5330	10660	26650	53300
cis-1,2-Dichloroethene	10.64	21.28	53.20	106.4	532.0	1064	5320	10640	26600	53200
Chloroform	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dichloroethane	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
1,1,1-Trichloroethane	10.74	21.48	53.70	107.4	537.0	1074	5370	10740	26850	53700
Benzene	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
Carbon Tetrachloride	10.55	21.10	52.75	105.5	527.5	1055	5275	10550	26375	52750
1,2-Dichloropropane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
Bromodichloromethane	10.66	21.32	53.30	106.6	533.0	1066	5330	10660	26650	53300
Trichloroethene	10.60	21.20	53.00	106.0	530.0	1060	5300	10600	26500	53000
1,4-Dioxane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
cis-1,3-Dichloropropene	11.16	22.32	55.80	111.6	558.0	1116	5580	11160	27900	55800
trans-1,3-Dichloropropene	10.64	21.28	53.20	106.4	532.0	1064	5320	10640	26600	53200
1,1,2-Trichloroethane	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Toluene	10.53	21.06	52.65	105.3	526.5	1053	5265	10530	26325	52650
Dibromochloromethane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
1,2-Dibromoethane	10.56	21.12	52.80	105.6	528.0	1056	5280	10560	26400	52800
Tetrachloroethene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Chlorobenzene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Ethylbenzene	10.55	21.10	52.75	105.5	527.5	1055	5275	10550	26375	52750
m,p-Xylenes	21.23	42.46	106.15	212.3	1061.5	2123	10615	21230	53075	106150
Styrene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
o-Xylene	10.54	21.08	52.70	105.4	527.0	1054	5270	10540	26350	52700
1,1,2,2-Tetrachloroethane	10.56	21.12	52.80	105.6	528.0	1056	5280	10560	26400	52800
1,3,5-Trimethylbenzene	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
1,2,4-Trimethylbenzene	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
1,3-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,4-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dibromo-3-chloropropane	10.53	21.06	52.65	105.3	526.5	1053	5265	10530	26325	52650
1,2,4-Trichlorobenzene	10.43	20.86	52.15	104.3	521.5	1043	5215	10430	26075	52150
Naphthalene	10.83	21.66	54.15	108.3	541.5	1083	5415	10830	27075	54150
Hexachloro-1,3-butadiene	10.59	21.18	52.95	105.9	529.5	1059	5295	10590	26475	52950

Note 1: The concentrations detailed in Table 3A may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	**25ng**	**Compound Name**	**25ng**	**Compound Name**	**25ng**
Bromochloromethane (IS1)	**12.5**	1,1,1-Trichloroethane	26.475	alpha-Pinene	26.575
Propene	26.275	Isopropyl acetate	53.050	n-Propylbenzene	26.725
Dichlorodifluoromethane (CFC 12)	26.250	1-Butanol	53.075	3-Ethyltoluene	26.550
Chloromethane	26.225	Benzene	26.525	4-Ethyltoluene	26.525
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	26.375	Carbon Tetrachloride	26.600	1,3,5-Trimethylbenzene	26.525
Vinyl Chloride	26.250	Cyclohexane	53.125	alpha-Methylstyrene	26.550
1,3-Butadiene	26.250	tert-Amyl Methyl Ether	26.525	2-Ethyltoluene	26.550
Bromomethane	26.250	1,2-Dichloropropane	26.525	1,2,4-Trimethylbenzene	26.525
Chloroethane	26.225	Bromodichloromethane	26.700	n-Decane	26.525
Ethanol	132.65	Trichloroethene	26.550	Benzyl Chloride	26.550
Acetonitrile	26.650	1,4-Dioxane	26.600	1,3-Dichlorobenzene	26.475
Acrolein	26.525	Isooctane	26.525	1,4-Dichlorobenzene	26.650
Acetone	133.05	Methyl Methacrylate	53.000	sec-Butylbenzene	26.550
Trichlorofluoromethane	26.275	n-Heptane	26.600	p-Isopropyltoluene	26.550
Isopropyl Alcohol	53.025	cis-1,3-Dichloropropene	26.275	1,2,3-Trimethylbenzene	26.550
Acrylonitrile	26.575	4-Methyl-2-Pentanone	26.575	1,2-Dichlorobenzene	26.550
1,1-Dichloroethene	26.575	trans-1,3-Dichloropropene	26.675	d-Limonene	26.550
tert-Butanol	53.275	1,1,2-Trichloroethane	26.525	1,2-Dibromo-3-Chloropropane	26.475
Methylene Chloride	26.550	**Chlorobenzene-d5 (IS3)**	**12.5**	n-Undecane	26.600
Allyl Chloride	26.500	**Toluene-d8 (S)**	**12.5**	1,2,4-Trichlorobenzene	26.500
Trichlorotrifluoroethane	26.450	Toluene	26.450	Naphthalene	26.700
Carbon Disulfide	26.675	2-Hexanone	26.575	n-Dodecane	26.550
trans-1,2-Dichloroethene	26.675	Dibromochloromethane	26.600	Hexachlorobutadiene	26.575
1,1-Dichloroethane	26.550	1,2-Dibromoethane	26.450	Methacrylonitrile	26.550
Methyl tert-Butyl Ether	26.600	Butyl Acetate	26.950	Cyclohexanone	26.575
Vinyl Acetate	132.55	n-Octane	26.500	tert-Butylbenzene	26.500
2-Butanone (MEK)	26.550	Tetrachloroethene	26.575	n-Butylbenzene	26.500
cis-1,2-Dichloroethene	26.475	Chlorobenzene	26.500		
Diisopropyl Ether	26.575	Ethylbenzene	26.450		
Ethyl Acetate	53.275	m- & p-Xylene	53.025		
n-Hexane	26.600	Bromoform	26.550		
Chloroform	26.475	Styrene	26.475		
1,2-Dichloroethane-d4 (S)	**12.5**	o-Xylene	26.450		
Tetrahydrofuran	26.575	n-Nonane	26.475		
Ethyl tert-Butyl Ether	26.525	1,1,2,2-Tetrachloroethane	26.500		
1,2-Dichloroethane	26.500	**4-Bromofluorobenzene (S)**	**12.5**		
1,4-Difluorobenzene(IS2)	**12.5**	Cumene	26.525		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	525.0
Chloromethane	524.5
Vinyl Chloride	525.0
1,3-Butadiene	525.0
Bromomethane	525.0
Chloroethane	524.5
Acrolein	530.5
Acetone	2661.0
Freon-11	525.5
1,1-Dichloroethene	531.5
Methylene Chloride	531.0
Freon-113	529.0
trans-1,2-Dichloroethene	533.5
1,1-Dichloroethane	531.0
Methyl tert-Butyl Ether	532.0
cis-1,2-Dichloroethene	529.5
Chloroform	529.5
1,2-Dichloroethane	530.0
1,1,1-Trichloroethane	529.5
Benzene	530.5
Carbon Tetrachloride	532.0
1,2-Dichloropropane	530.5
Bromodichloromethane	534.0
Trichloroethene	531.0
1,4-Dioxane*	532.0
cis-1,3-Dichloropropene	525.5
trans-1,3-Dichloropropene	533.5
1,1,2-Trichloroethane	530.5
Toluene	529.0
Dibromochloromethane	532.0
1,2-Dibromoethane	529.0
Tetrachloroethene	531.5
Chlorobenzene	530.0
Ethylbenzene	529.0
m,p-Xylenes	1060.5
Styrene	529.5
o-Xylene	529.0
1,1,2,2-Tetrachloroethane	530.0
1,3,5-Trimethylbenzene	530.5
1,2,4-Trimethylbenzene	530.5
1,3-Dichlorobenzene	529.5
1,4-Dichlorobenzene	533.0
1,2-Dichlorobenzene	531.0
1,2-Dibromo-3-chloropropane	529.5
1,2,4-Trichlorobenzene	530.0
Naphthalene	534.0
Hexachloro-1,3-butadiene	531.5

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee ________________ Trainer _________________ Instrument _____ Training Completion Date ____________

1. Read SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date _________

2. Read Methods TO-14A & TO-15A *Training Duration* ______________ Trainer ___ Trainee ___ Date _________

3. Demonstrated understanding of the scientific basis of the analysis Trainer ___ Trainee ___ Date _________
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* ____________________
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ___ Trainee ___ Date _________
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* ____________________
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ___

5. Observe performance of SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date _________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* ______________ Trainer ___ Trainee ___ Date _________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date _________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ___ Trainee ___ Date _________
   ___autosampler *Training Duration* __________________
   ___GC and capillary column installation *Training Duration* __________________
   ___mass spectrometer *Training Duration* __________________
   ___data system *Training Duration* __________________

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:______________________ ICAL ID:____________________ LIMS ICAL ID:____________________

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation in the ICAL file? .... ☐
- ☐ BFB Tune analysis Report .... ☐
- ☐ Calibration Status Report (aka Calibration History) .... ☐
- ☐ Response Factor Report/Percent RSD .... ☐
- ☐ Quant Report for each calibration std (including manual integration documentation) .... ☐
- ☐ ICV Quantitation Report .... ☐
- ☐ TO-15 Standard Calculation Spreadsheet .... ☐

☐ 2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)? .... ☐

☐ 3. Have all the calibration standards been analyzed within 24 hours of each other? .... ☐

☐ 4. Does the BFB tune check standard analysis at the start meet the tune criteria? .... ☐

☐ 5. Are all the analytes in the blank analysis <MRL? .... ☐

☐ 6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels? .... ☐

☐ 7. Were the standards analyzed from low concentration to high concentration? .... ☐

☐ 8. For each analyte, are there no levels skipped? .... ☐

☐ 9. For each analyte, is there only one value used for each calibration level? .... ☐

☐ 10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL? .... ☐

☐ 11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve? .... ☐

☐ 12. For each analyte, are the corresponding upper levels free from saturation? .... ☐

☐ 13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason? .... ☐

☐ 14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%? .... ☐

☐ 15. DoD/Navy: Is the average RSD ≤30% for all analytes? .... ☐

☐ 16. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard? .... ☐

☐ 17. Percent recovery for each analyte in the ICV 70%-130% (AZ: 50-150% for VA)? .... ☐

☐ 18. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound? .... ☐

☐ 19. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard? .... ☐

☐ 20. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages. .... ☐

☐ 21. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds?
☐ Yes ☐ No Note exceptions and corresponding MRLs below – *Specify applicable range...* .... ☐

☐ 22. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)? .... ☐

COMMENTS:

Analyst: ______________________________ Secondary Reviewer: ______________________________

Attachment 3

Daily QC and Sample Review Checklists

EPA Compendium Method TO-15 - Daily QC Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** ____________________

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation present? .... ☐
  ☐ CORRECT BFB Tune analysis Report
  ☐ CCV analysis Quantitation Report & %D Report
  ☐ LCS analysis Quantitation Report
  ☐ MB analysis Quantitation Report

☐ 2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above? .... ☐

☐ 3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**? .... ☐

☐ 4. Does the **CCV** have a difference ≤30% for all analytes? .... ☐
[Note all outliers biased high and/or low]

☐ 5. **DoD**: Does the **Closing CCV** have a difference ≤30% for all analytes? .... ☐
[Note all outliers biased high and/or low]

☐ 6. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)? .... ☐

☐ 7. All **IS** responses within ±40% of CCV or the midpoint in the ICAL? .... ☐

☐ 8. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%) .... ☐

☐ 9. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)? .... ☐

☐ 10. **LCS** %R within lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)? .... ☐

☐ 11. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits? .... ☐

☐ 12. **DoD/Navy**: **DLCS** analyzed? .... ☐

Air-Phase Petroleum Hydrocarbons

☐ 1. Does the **CCV** meet the following criteria? .... ☐
- Percent difference ≤30%.
- One compound or range can be >30%, but less than 50%.
- No single analyte or range may be >50%.

[Note outliers biased high and/or low in comments below]

☐ 2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if: .... ☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

☐ 3. Are the analytes in the **LCS** within 70%-130% recovery? .... ☐

COMMENTS:

Analyst/LIMS Run Approval: ______________________ Secondary/LIMS Supervisor Approval: ______________________

EPA Compendium Method TO-15 - Sample Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _______________ **Project #:** _______________

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. All analyte hits in the samples within the **calibration range** and/or noted? .... ☐
☐ 2. All **peak integrations** acceptable? .... ☐
☐ 3. All **manual integrations** flagged and documented? .... ☐
☐ 4. Have **Q values** been verified for each peak? .... ☐
☐ 5. All **calculations** correct? .... ☐
☐ 6. Has the analyst initialed and dated each **quantitation report**? .... ☐
☐ 7. For **TICs** are the relative intensity and other requirements met (associated MB reported)? .... ☐
☐ 8. **Auto report** correct? .... ☐
☐ 9. **MRL** = _______ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng) .... ☐
☐ 10. Pressurized with **Helium**? Is the worksheet completed for all samples? .... ☐
☐ 11. Report to **MDL**? ☐ Yes ☐ No .... ☐
☐ 12. **Global Minimum Detection Limit** = _______ ☐ ng ☐ pg .... ☐
☐ 13. **DOD:** Are **manual integrations** notated in the **case narrative?** .... ☐

Air-Phase Petroleum Hydrocarbons

☐ 1. Are all manual **integrations** flagged and documented (except for HC ranges)? .... ☐
☐ 2. Are the associated ICAL responses correct? .... ☐
☐ 3. Are the sample responses entered into the template correctly? .... ☐
☐ 4. Are the TO-15 target compounds entered into the template correctly? .... ☐
☐ 5. Does the lab **duplicate** meet RPD ≤30% for results >5x the MRL? Otherwise, repeat analyses if: .... ☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐ 1. CASE NARRATIVE COMPLETED? .... ☐

Analyst/LIMS Run Approval: ______________________________ Secondary/LIMS Supervisor Approval: ______________________________

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAP 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

Attachment 5

Tekmar AutoCan Trap Packing Instructions

Tekmar AutoCan Trap Packing Instructions

The internal sample trap on the AutoCan is a 1/8" x 12" thin-walled stainless steel tube, usually coated with fused silica (Silcosteel). It is packed with a combination of graphitized carbon black and carbon molecular sieve adsorbents, with the weakest adsorbent at the top (inlet) and the strongest at the bottom (outlet). Each bed is separated by a small plug of untreated glass wool. Untreated is used because DCMS-treated wool will release siloxanes when heated to the temperatures used for TO-15 analysis.

The adsorbents listed below are further refined at the lab by sifting in an 80-mesh sieve. This removes the smaller particles and leaves a very uniform product of about 60-mesh size. Getting rid of the "fines" helps ensure good flow through the trap during sampling and reduces the pressure drop across the trap. A tightly-packed trap can lead to problems such as poor reproducibility, slowed flow rates, and channeling (small spaces in the beds that let analytes pass through).

Adsorbent	Mesh	Supplier	Catalog #	Packing Amount (mg)
Carbosieve SIII	60/80	Supelco	10184	40
Carbosieve G	60/80	Supelco	10198	30
Carbopack Z	60/80	Supelco	20273	30
Tenax TA	20/30 or 45/60	Supelco	10257	rest of trap

Old traps can be reused if unpacked carefully and cleaned and baked out properly. Use a glass wool puller to remove the wool plugs, and gently tap the sorbent out onto a piece of paper. If necessary, use the other end of the puller to loosen the sorbent bed, being careful not to scratch the inside of the trap. Discard the old sorbent. Rinse the empty trap with methanol, then bake in a GC oven for 30 minutes at 150°C.

The total length of the adsorbent bed is 12 to 13cm. You want to leave 2 to 3cm of space above the top of the last glass wool layer to ensure that all of the material is within the heated zone of the AutoCan trap heater.

With clean hands (no lotion!) place a small amount of glass wool, about 10-15mg, into the top of the trap and work it in with a piece of wire or tubing. Then use the trap packing tool (the larger steel rod that just barely fits inside the trap) to hold the plug in the trap while you pull away any loose strands of wool. Then use the long steel tube to push the plug down about 15cm. The idea is to keep the plug very compact, so it is a good idea to use the trap packing tool to push up from the bottom while pushing the wool in from the top, meeting 15cm down. The plug should not move too easily when pushed.

Weigh out the first sorbent (Carbosieve SIII) on weighing paper using the analytical balance. Using the glass funnel and a short piece of silicone tubing, pour the sorbent into the top of the trap. Tap on counter to get it all out of the funnel, then remove the funnel and tap some more to settle the sorbent into a compact bed. It is very important that there are no air spaces in the bed. However, it is also very important not to compress the sorbents too much, so be very careful when placing the glass wool plugs.

Place a glass wool plug on top of the first bed, starting as described above for the first plug. Push it gently onto the top of the sorbent with very little pressure.

Proceed with the other three packings in the table above (Carbosieve G, Carbopack Z, and Tenax TA).

After placing the last glass wool plug on top, turn the trap over and gently tap it on a piece of white paper to see if any sorbent comes out. If it does, you need to add more glass wool.

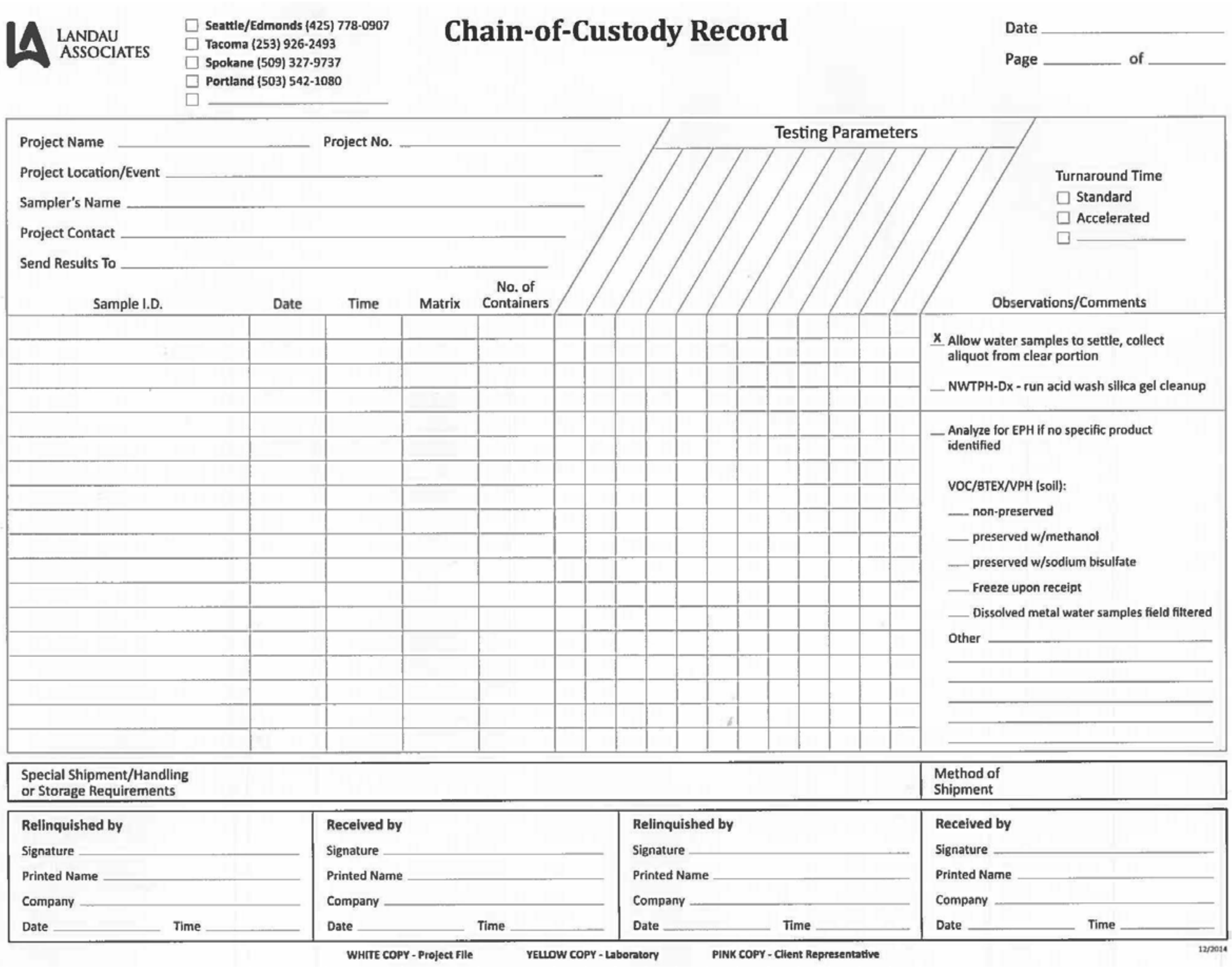
Now the trap needs to be conditioned in the trap heater. The sorbent manufacturers recommend that they be conditioned at succeedingly higher temperatures, with the final temperature being about 20-30˚C higher than the desorb temperature. The reason is that the sieves hold a lot of air and moisture and it is better to drive these off at lower temperatures to avoid damage to the material, such as cracking and oxidation which creates active sites. The temperatures and times are:

80˚C for 30 minutes, 50 to 100ml/min nitrogen or helium flow
200˚C for 30 minutes
265˚C for at least 3 hours

These temperatures are set using the variable power controller and thermocouple meter. Repeat for the other temperatures (low to high). Make sure the gas toggle valve in back is open, and measure flow at the top of the trap.

ATTACHMENT B-2

Example Field Forms



LANDAU ASSOCIATES

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☐ Tacoma (253) 926-2493
☐ Spokane (509) 327-9737
☐ Portland (503) 542-1080
☐ ______________

Chain-of-Custody Record

Date ______________
Page ________ of ________

Project Name ______________ Project No. ______________
Project Location/Event ______________
Sampler's Name ______________
Project Contact ______________
Send Results To ______________

Testing Parameters

Turnaround Time
☐ Standard
☐ Accelerated
☐ ______________

Sample I.D.	Date	Time	Matrix	No. of Containers												

Observations/Comments

X Allow water samples to settle, collect aliquot from clear portion
__ NWTPH-Dx - run acid wash silica gel cleanup
__ Analyze for EPH if no specific product identified

VOC/BTEX/VPH (soil):
__ non-preserved
__ preserved w/methanol
__ preserved w/sodium bisulfate
__ Freeze upon receipt
__ Dissolved metal water samples field filtered
Other ______________

Special Shipment/Handling or Storage Requirements

Method of Shipment

Relinquished by	Received by	Relinquished by	Received by
Signature ______	Signature ______	Signature ______	Signature ______
Printed Name ______	Printed Name ______	Printed Name ______	Printed Name ______
Company ______	Company ______	Company ______	Company ______
Date ______ Time ______	Date ______ Time ______	Date ______ Time ______	Date ______ Time ______

WHITE COPY - Project File YELLOW COPY - Laboratory PINK COPY - Client Representative

12/2014

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TECT Aerospace
Everett, Washington

Sample Chain-of-Custody Form

Figure 1

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Field Report

Project No.: ______ Report No.: ______

Client: ______ Date: ______

Project Name: ______ DPD Permit No.: ______

Location: ______

Weather Conditions: ______

Prepared By: ______

Visitors: ______

Unsatisfactory Conditions & Recommended Correction: ______

Attachments: ______

Signed: ______

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Groundwater Low-Flow Sample Collection Form

Project Name: ____________________ Project Number: ____________________
Event: ____________________ Date/Time: ____________________
Sample Number: ____________________ Weather: ____________________
Landau Representative: ____________________

WATER LEVEL/WELL/PURGE DATA

Well Condition: Secure (YES or NO) Damaged (YES or NO) Describe: ____________________
DTW Before Purging (ft) __________ Time: __________ Flow through cell vol. __________ GW Meter No.(s) __________
Begin Purge: Date/Time: __________ End Purge: Date/Time: __________ Gallons Purged: __________
Purge water disposed to: ☐ 55-gal Drum ☐ Storage Tank ☐ Ground ☐ Other __________

Time	**Temp (°F/°C)**	**Cond. (uS/cm)**	**D.O. (mg/L)**	**pH**	**ORP (mV)**	**Turbidity (NTU)**	**DTW (ft)**	**Internal Purge Volume (gal)**	**Comments/ Observations**
Purge Goals: Stablization of Parameters for three consecutive readings within the following limits	**+/- 3%**	**+/- 3%**	**+/- 10%**	**+/- 0.1 units**	**+/- 10 mV**	**+/- 10%**	**< 0.3 ft**	**>/= 1 flow through cell**	

SAMPLE COLLECTION DATA

Sample Collected With: ☐ Bailer ☐ Pump/Pump Type __________ __________
Made of: ☐ Stainless Steel ☐ PVC ☐ Teflon ☐ Polyethylene ☐ Other ☐ Dedicated
Decon Procedure: ☐ Alconox Wash ☐ Tap Rinse ☐ DI Water ☐ Dedicated
(By Numerical Order) ☐ Other __________
Sample Description (color, turbidity, odor, sheen, etc.): ____________________

Replicate	**Temp (°F/°C)**	**Cond. (uS/cm)**	**D.O. (mg/L)**	**pH**	**ORP (mV)**	**Turbidity (NTU)**	**DTW (ft)**	**Ferrous iron (Fe II)**	**Comments/ Observations**
1									
2									
3									
4									
Average:									

QUANTITY	**TYPICAL ANALYSIS ALLOWED PER BOTTLE TYPE (Circle applicable or write non-standard analysis below)**
	(8260) (8010) (8020) (NWTPH-G) (NWTPH-Gx) (BTEX) WA ☐ OR ☐
	(8270) (PAH) (NWTPH-D) (NWTPH-Dx) (TPH-HCID) (8081) (8141) (Oil & Grease) WA ☐ OR ☐
	(pH) (Conductivity) (TDS) (TSS) (BOD) (Turbidity) (Alkalinity) (HCO3/CO3) (Cl) (SO4) (NO3) (NO2) (F)
	(COD) (TOC) (Total PO4) (Total Kiedahl Nitrogen) (NH3) (NO3/NO2)
	(Total Cyanide) (WAD Cyanide) (Free Cyanide)
	(Total Metals) (As) (Sb) (Ba) (Be) (Ca) (Cd) (Co) (Cr) (Cu) (Fe) (Pb) (Mg) (Mn) (Ni) (Ag) (Se) (Tl) (V) (Zn) (Hg) (K) (Na)
	(Dissolved Metals) (As) (Sb) (Ba) (Be) (Ca) (Cd) (Co) (Cr) (Cu) (Fe) (Pb) (Mg) (Mn) (Ni) (Ag) (Se) (Tl) (V) (Zn) (Hg) (K) (Na) (Hardness) (Silic
	VOC (Boeing short list)
	Methane Ethane Ethene Acetylene
	others

Duplicate Sample No(s): ____________________
Comments: ____________________
Signature: ____________________ Date: __________

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Exploration No. ____________________
Date __________ Hour _____________

Log of Exploration

Project Name ______________________ Project No. ______________________

Client/owner ______________________ Exploration Operator ______________

Exploration Method ____

____

Logged by ________________________ Exploration Completed ______________

Ground Surface Conditions ____

Weather Conditions ____

Location Sketch (show dimensions to mapped features)

○ North Arrow

(East) (North)
Coordinates: "x" __________ "y" __________ Method __________

Elevations ______________________ Datum ________________

Sampler and Hammer Information

a = 3.25-in. O.D. – D&M	1 = 300-lb./30-in. Drop
b = 2.0-in. O.D. – SPT	2 = 140-lb./30-in. Drop
c = Shelby Tube	3 = Pushed
d = Grab Sample	4 = Vibrocore
g = 2.5-in. O.D.– WSDOT	5 = ______________
h = 3.0-in. O.D. – M.Calif.	
i = _______________	

Water Level Information

Date		
Time		
Depth to Water		
Hole Depth		
Casing Depth		

Sample Depth (top) (ft.)	Sample Length (ft.)	Recovery Length (ft.)	Retained Depth (top) (ft.)	Retained Length (ft.)	Sample Number	Sampler/Hammer Codes	Blow Counts	Other Test Data) ____	USCS Symbol / Unit Contact	Depth Scale (ft)	Sample Description Color, secondary soil type, PRIMARY SOIL TYPE with modifiers and minor components (density/consistency, moisture)(geologic unit)	Comments on Heave, Water Conditions, & Drilling Action
										0		
										1		
										2		
										3		
										4		
										5		
										6		
										7		
										8		
										9		
										0		
										1		
										2		
										3		
										4		
										5		
										6		
										7		
										8		
										9		
										0		

Total Depth _____________ Finish Date _______________ Hour _____________ Continued

Exploration No.____________________

Date ___________ Hour_________

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Log of Test Pit

Project Name ______________________ Project No. ________________________

Client/owner _______________________ Exploration Operator ________________

Logged by ________________________ Exploration Completed _______________

Ground Surface Conditions ____

Weather Conditions ____

Location Sketch (show dimensions to mapped features)

○ North Arrow

Groundwater seepage:
none slow moderate rapid
@ _____________ feet

(East) (North)
Coordinates: "x" _________ "y" _________ Method ___________

Elevations _____________________ Datum _________________

Sample Description
Color, secondary soil type, PRIMARY SOIL TYPE with modifiers and minor components
(density/consistency, moisture)(geologic unit)

Length of Test Pit (ft.) (View Direction) ______________

Sample Depth (top) (ft.)	Sample Length (ft.)	Sample Number	(Other Test Data) ____	USCS Symbol	Depth Scale (ft)
					0

Total Depth ____________ Finish Date ____________ Hour ____________

Comments/Field Notes: ____

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PROJECT ____________ PROJECT NO. ________

EVENT ____________

SAMPLE NO. ____________

DATE COLLECTED ________ TIME ________

Soil/Sediment Sample Collection Form

Weather ____________ Collector(s) ____________

SAMPLE LOCATION/COMPOSITE DATA

Sample Type: ○ Soil ○ Sediment ○ Other ____________

Sample Location: ____________

Sample Composited: ○ Horizontally Locations: ____________

○ Vertically Depth Ranges: ____________

○ Not Composited ○ Other: ____________

Elevation and Reference: ____________

SAMPLE COLLECTION DATA

Sample Collected From: ○ Hand-Dug Hole ○ Test Pit ○ Boring ○ Catch Basin/Manhole ○ Other ________

Sample Collected With: ○ Bowl ○ Spoon ○ Split Barrel ○ Shovel ○ Auger ○ Other ________

Made of: ○ Stainless Steel ○ Steel ○ Other ________

Decon Procedure: ○ Alconox Wash ○ Tap Rinse ○ DI Water Rinse ○ Other ________ ○ Other ________

(By Numerical Order) ○ Other ____________

SAMPLE DESCRIPTION (color, grain size, density, moisture, etc.): ____________

SIZE	QUANTITY	TYPE	LABORATORY ANALYSIS
____	____	○ Glass ○ Plastic ○ Other ________	____________
____	____	○ Glass ○ Plastic ○ Other ________	____________
____	____	○ Glass ○ Plastic ○ Other ________	____________

Co-Located/Duplicate Sample No(s). ____________

Photo No. ____________ Roll No. ____________

Comments: ____________

○ *Continued on Back*

Signature ____________ Date ________

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Survey Field Notes Form

Project Name: ______________________________

Project Number: ___________________________

Location: _________________________________

Date: _____________________________________

Client: ___________________________________

Landau Rep: ______________________________

Station	$B^{(+)}$ s	H I	$F^{(-)}$ s	Elevation	Description/Comments

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As-Built Well Completion Form

Exploration No.: ____________________

Well No. (If different than Expl. No.): ________

Client/Owner: ______________ Project No.: ______________

Project Name: ______________________________

Drilling Co.: ______________________________

LAI Rep(s): ______________________________

Installation Start Date: ______________ Hour: ________

Installation Finish Date: ______________ Hour: ________

Well Type: ☐ Single ☐ Nested ☐ Clustered

BORING AND WELL DIMENSIONS AND INSTALLATION DETAILS

DOE Unique Well No.: ______________________

Number of Pipes in Boring: ______________________

Boring Diameter at Top of Hole: ______________________

Does Diameter of Hole Change? ______________________

Boring Diameter at First Step Down: ______________

Depth of First Step Down: ______________

Boring Diameter at Second Step Down: ______________

Depth of Second Step Down: ______________

Well Completion Date: ______________________

Elevation of Well Cover: ______________________

Elevation of Top of Well Pipe: ______________________

Depth to Water: ______________________

Date: ______________ Time: ______________

____

____

MATERIALS USED

________ Sacks of ________________ Sand

________ Sacks of ________________ Concrete/Cement

________ Sacks of ________________ Grout Mix Used

________ Sacks of Bentonite Chips

________ Feet of ______-inch PVC Blank Casing

________ Feet of ______-inch PVC Slotted Screen

________ Threaded End Cap

________ Waterproof Well Seal/Slip Cap

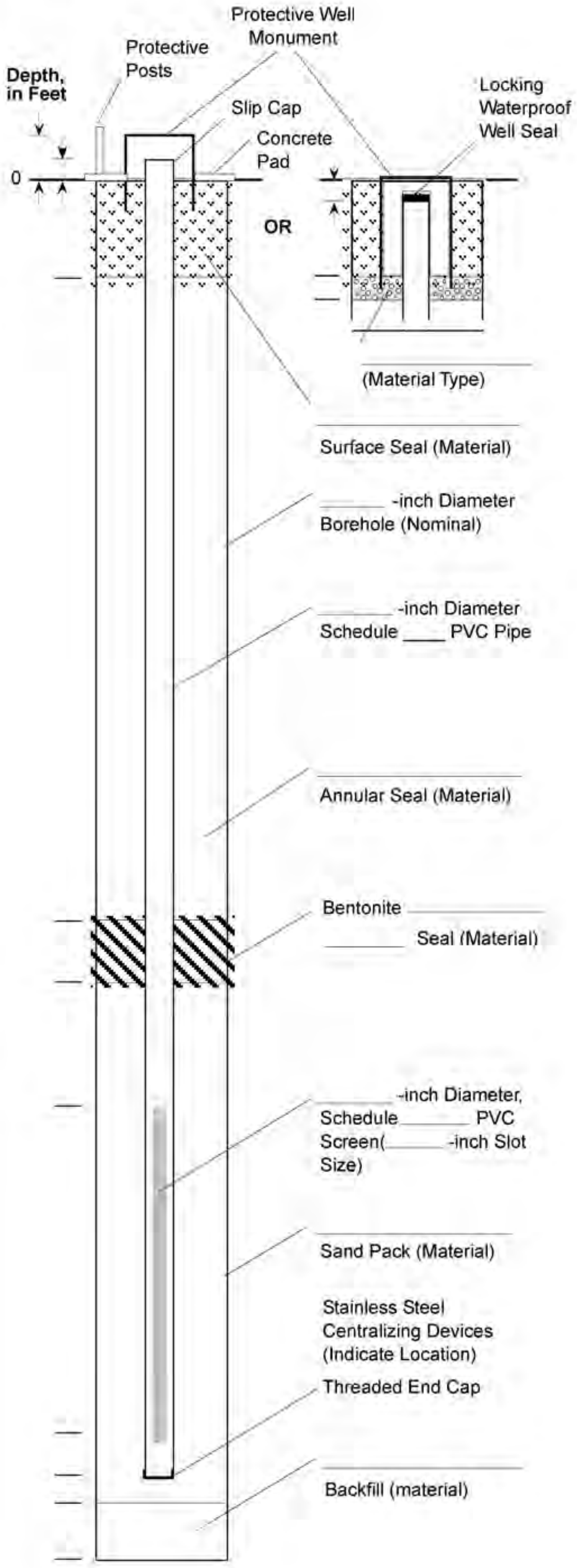
________ Flush Mount/Aboveground Protective Monument

________ Protective Posts

____

____

____



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Well Development Record

Project Name: ____________________
Location: ____________________
Client: ____________________

Project No. ____________________
Date: ____________________
Landau Representative: ____________________

Well Number: ____________________

Time: ____________________

Depth to Water: ____________________
Well Depth: ____________________
Casing Diameter: ____________________
Casing Volume: ____________________

Volume of Schedule 40 PVC Pipe

Diameter (inch)	O.D. (inch)	I.D. (inch)	Volume (gal/ln ft)	Wt. Water (lbs/ln ft)
1.25	1.660	1.380	0.08	0.64
2	2.375	2.067	0.17	1.45
4	4.500	4.026	0.66	5.51
6			1.47	12.24

Est. Purge Volume: ____________________

Method of Development: ____________________

Surge Block: Yes ______ No ______

Begin Development: Time:
Finish Development: Time:

Final Volume Purged: ____________________
Water Disposal: ☐ 55-gal drum ☐ Storage Tank ☐ Ground ☐ Other ________

Initial Water Quality: (Turbidity, Color, Odor, Other) ____________________ Initial Yield: ______
pH: ______ Temp: ______ Conductivity: ______ Turbidity: ___
Notes: ____________________

Water Quality Notes: ____________________

Gallons	pH	Temperature	Conductivity	Turbidity	Comments

Final Water Quality: (Turbidity, Color, Odor, Other) ____________________
Final Yield: ____________________
pH: ______ Temperature: ______ Conductivity: ______ Turbidity: ______

Depth to Water After Development: ____________________ Well Depth After Development: ____________________

APPENDIX C

Health and Safety Plan