Earth Systems
Environmental Engineering

August 17, 2016

Andrew Park
Hazardous Waste Programs Branch
U.S. Environmental Protection Agency Region 2
290 Broadway, 22nd Fl.
New York, NY 10007-1866

Re: Remedial Investigation Work Plan
AOC 12: Smith Creek
Hess Corporation Former Port Reading Complex (HC-PR)
835 West Avenue
Port Reading, Middlesex County, New Jersey
Program Interest No. 006148
NJDEP ISRA Case No. E20130449
EPA ID No. NJD045445483

Dear Andrew:

Attached please find the Remedial Investigation Work Plan (disc) for the above-referenced site. Please feel free to contact me at (732) 739-6444 if you have any questions or require additional information.

Sincerely,
EARTH SYSTEMS

John S. Virgie LSRP, PG
Senior Client Manager

cc:

Mr. Phil Cole, Case Manager (including 3 paper copies) – NJDEP
Mr. John Schenkewitz, Manager, Remediation - Hess Corporation
Ms. Krista Snyder, Manager, Remediation – Buckeye Partners, L.P.
Mr. Rick Ofsanko – Earth Systems

REMEDIAL INVESTIGATION WORKPLAN

AOC 12: Smith Creek and Detention Basin
Hess Corporation – Former Port Reading Complex
(HC-PR)
750 Cliff Road,
Port Reading, Middlesex County, New Jersey
NJDEP PI# 006148
ISRA Case No. E20130449
EPA ID No. NJD045445483

August 2016

Prepared for:

Hess Corporation

Trenton-Mercer Airport
601 Jack Stephan Way
West Trenton, New Jersey 08628

Prepared By:

Earth Systems
Environmental Engineering

1625 Route 71
Belmar, New Jersey 07719

REMEDIAL INVESTIGATION WORKPLAN

AOC 12: Smith Creek and Detention Basin
Hess Corporation – Former Port Reading Complex (HC-PR)
750 Cliff Road
Port Reading, Middlesex County, New Jersey
NJDEP PI# 006148

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1.0 INTRODUCTION

On behalf of Hess Corporation (Hess), Earth Systems, Inc. (Earth Systems) has prepared this Remedial Investigation Workplan (RIW) for the environmental Area of Concern (AOC) designated as AOC 12: Smith Creek and Detention Basin (AOC 12) at the Hess Corporation Former Port Reading Complex (HC-PR), located at 750 Cliff Road, in Port Reading (Woodbridge Township), Middlesex County, New Jersey (the Site).

A United States Geological Survey (USGS) 7.5 minute series quadrangle map (Arthur Kill, New Jersey), depicting the HC-PR facility and associated land features is presented as **Figure 1** and the location of AOC 12 and the associated groundwater monitoring wells is presented as **Figure 2**.

Due to historic operations, the Site is jointly regulated by both the New Jersey Department of Environmental Protection (NJDEP) and the Environmental Protection Agency (EPA). The NJDEP Industrial Site Recovery Act (ISRA) was triggered when HC-PR executed an agreement to sell the Port Reading Complex. The Site is regulated under EPA's Resource Conservation and Recovery Act (RCRA) since former operations at the Site required the treatment, storage, and disposal of hazardous waste.

In accordance with the New Jersey Technical Requirements for Site Remediation (7:26E-4.1d), this RIW is being submitted for approval since the Site is regulated under RCRA, in addition to being subject to reporting requirements under ISRA. This RIW is an AOC specific plan solely intended to address investigation of AOC 12: Smith Creek and Detention Basin. A RIW was previously submitted by Delta Consultants in September 2007 for AOC 12. However, the work plan was never implemented. This RIW supersedes the previous RIW prepared and submitted by Delta Consultants.

The following RIW provides a summary of historic groundwater investigation activities and the potential impacts associated with AOC 12. Following the summary are recommendations for additional investigation activities to delineate impacts in all effected media at the Site to satisfy New Jersey Department of Environmental Protection (NJDEP) requirements in accordance with the New Jersey Administrative Code (N.J.A.C.) 7:26E, *The Technical Requirements for Site Remediation (TRSR)*; N.J.A.C 7:26C, *The Administrative Requirements for the Remediation of Contaminated Sites (ARRCS)*; N.J.S.A. 58:10C-1 et seq., *The Site Remediation Reform Act (SRRA)*; and the associated NJDEP SRRA Guidance Documents. All information obtained during the proposed remedial activities will be documented in an AOC 12 Remedial Investigation Report (RIR), the site-wide Ecological Risk Assessment (ERA), and the CA725 and CA750 EPA Environmental Indicator (EI) Reports.

2.0 BACKGROUND

2.1 Site Description

The HC-PR facility is an approximate 223-acre irregularly shaped parcel, situated in an industrially developed waterfront area. A Site location map for the HC-PR facility is presented as **Figure 1**. The HC-PR facility is identified as Block 756, Lot 3; Block 756.01, Lots 1.02, 2, and 3; Block 756.02, Lots 1 and 8; Block 757, Lot 1; Block 760, Lot 6; Block 760.01, Lots 2 and 3; Block 760.02, Lots 1, 2, and 3; Block 1096.01, Lot 6, and Block 664.01, Lots 1.01 and 1.02**.**

The HC-PR facility is located east of Cliff Road and abuts the southern property boundary of the Conrail Port Reading Rail yard. Immediately east-southeast of the facility is the Arthur Kill shipping channel,

and to the southwest is the PSE&G Sewaren Generating facility. The former Port Reading Coal Docks, currently owned by Prologis Corporation, are located to the northeast. Port Reading Avenue is located to the northwest. A mixture of industrial and commercial properties are located to the west. Two (2) residential properties are located up-gradient to the northwest, and an industrial property is located to the south.

The HC-PR facility formerly processed low sulfur gas oils and residuals as feed to a Fluidized Catalytic Cracking Unit (FCCU) that converted gas oil into gasoline, fuel oil, and other hydrocarbon products (e.g. methane, ethane and liquid petroleum gas). The HC-PR site operations were initiated in 1958 with a Crude Topping Unit and underwent various expansions between 1958 and 1970. In 1974, refining operations were suspended and the facility operated only as a bulk storage and distribution terminal until 1985. In April 1985, following a retrofit, the HC-PR facility resumed refining operations. The refinery was demolished in 2015, and currently the Site is operated only as a bulk storage and distribution terminal.

A detailed description of AOC 12 is included as **Section 3.2.**

2.2 Site Geology and Hydrogeology

The geology of the HC-PR facility was determined from the data collected at the HC-PR facility, during the subsurface investigations, and from the Geologic Map of the State of New Jersey. The HC-PR facility is underlain by the Magothy and Raritan formations, which are the lowest members of the Cretaceous-age Coastal Plain physiographic sediments. The Raritan Formation consists of sands and clays of variable color and grain size, and the overlying Magothy Formation consists of dark lignitic sand and clay containing glauconite near the top. The western section of the HC-PR facility is underlain by a thick clay unit, while marsh deposits underlie the eastern and southeastern section of the HC-PR facility.

The shallow unconfined water table at the HC-PR facility was encountered between approximately 2 and 11 feet below ground surface (bgs). Groundwater flow is predominately southeasterly in the northwest portion of the HC-PR facility and east-southeasterly in the central portion of the HC-PR facility. The HC-PR facility wells located adjacent the Arthur Kill and North Drainage Ditch are affected by tidal influences. Wells located further away from the Arthur Kill are generally unaffected by tidal influence. An average hydraulic gradient of approximately 0.001 feet /per feet was calculated for the Site. The general flow of ground water is toward the south-southeast in the vicinity of AOC 12.

2.3 Topography and Surface Water

Topography of the Site and surrounding area is generally flat with a very gradual slope downward toward the Arthur Kill. The total difference in topographic relief on the developed portion of the site is less than 5 feet. Surveyed ground surface elevations indicated that the developed portion of the property, which has an approximate total area of 223 acres, ranges in elevation from 5 to 10 feet above MSL referenced to North American Vertical Datum of 1988 (NAVD88).

A detention basin (AOC 12) is located on the southern portion of the Site. Stormwater enters the detention basin through both overland flow as well as Site drainage outfalls that historically discharged directly to the basin. Outfalls that historically were routed to the detention basin were capped with concrete.

Smith Creek is currently located offsite to the south. Smith Creek flows to the south and discharges directly to the Arthur Kill, which is adjacent to the Site on the eastern side.

A more detailed description of the history and current status of the detention basin and Smith Creek is included in **Section 3.0.**

3.0 AOC 12: SMITH CREEK AND DETENTION BASIN

3.1 History

HC-PR began operations on the Site in 1958. Based on a review of historic aerial photographs, it appears that the northern portion of the Creek and its tributaries were filled between 1954 and 1957. By 1963, only the southern portion of Smith Creek was present on the Site. All northern portions of the Creek were filled-in, excluding the northern drainage ditch. The detention basin present on the Site was created between 1966 and 1969. In 1972, Smith Creek was no longer present on the Site and the detention basin was no longer directly connected to the off-site portion of Smith Creek. Between 1980 and 1986, the detention basin appears to be slightly smaller in size in comparison to previous historic aerial photographs.

The following provides a brief summary of historic releases and site impacts, which had the potential to directly impact both the detention basin and Smith Creek.

- **1969 Spill (HS-1 91-1-28-1002-17)**
  In October 1969, a 200,000-barrel crude oil storage tank failure occurred at the facility and approximately eight (8) million gallons of crude oil were released onto the adjacent tank field which was surrounded by a retaining berm. According to historic records, the majority of the crude oil was contained within the bermed area and recovered. However, a seep in the berm allowed an undetermined amount of oil to flow into the on-site retention basin and subsequently into Smith Creek. Crude oil was identified within Smith Creek and remediated via the use of absorbent booms and vacuum extraction.

- **1991 Spill (HS-4 91-9-25-1014-00)**
  On September 25, 1991, approximately 500 to 700 gallons of oil overflowed the American Petroleum Institute (API) separator due to heavy rainfall. Historic spill records indicated that oil was observed in Smith Creek. However, based on the distance from the API separator to Smith Creek, it is assumed that oil was actually observed in the on-site detention basin, which was the former location of Smith Creek. The spill was addressed via vacuum recovery and absorbent booms.

- **Former Site Operations**
  The detention basin and Smith Creek are downgradient from several known areas of impacts on the Site. Based on the interpreted southerly-southeasterly groundwater flow direction, AOC 12 is situated downgradient from both AOC 2: South Landfarm and AOC 10: Truck Loading Rack. Impacts associated with these AOCs generally consist of isolated areas of free product as well as groundwater impacts from volatile organic compounds (VOCs), metals, and low levels of semi-volatile organic compounds (SVOCs). The former portions of Smith Creek and tributaries that were filled as part of Site improvements could be functioning as preferential pathways channeling impacted groundwater toward the detention basin.

3.2 Description of Current Conditions

The detention basin is approximately 800 feet by 600 feet in size with a depth of approximately 5 to 6 feet. Contaminant migration pathways to the detention basin include groundwater and overland flow. Outfalls that historically were routed to the detention basin were capped with concrete. Contaminant migration pathways away from the detention basin include groundwater and overland flow. Phragmites dominated wetlands and herbaceous wetlands are present adjacent to the detention basin.

The only remaining portion of Smith Creek that remains on-site is the Northern Drainage Ditch. The Northern Drainage Ditch will be addressed as part of closure of the No. 1 Landfarm and is not addressed in this RIW.

Utilizing tools provided by NJDEP's Geoweb, Smith Creek is approximately 125-175 feet wide and approximately 5,000 feet long from where the Creek begins, to where it eventually discharges into the Arthur Kill.

Directly to the north of Smith Creek is a pond (also located off-site) that is connected to Smith Creek via piping. During the RIW activities proposed below, a thorough inspection of the Pond will be conducted.

4.0 SUMMARY OF AOC 12 GROUNDWATER INVESTIGATION ACTIVITIES

Groundwater investigation activities to address AOC 12 began in 2002. However, the majority of the monitoring wells were installed in 2013 or later. Monitoring wells have been installed to monitor shallow, intermediate, and deep groundwater zones. The following groundwater monitoring wells have been used to assess groundwater conditions associated with AOC 12:

Monitoring Well ID	Date Installed	Well Location	Well Depth (from TOC)	Screened Interval/Groundwater Interval
PER-2	4/10/2002	Side-gradient	9 ft	2-9 ft (shallow)
PER-2D	5/2/2013	Side-gradient	30 ft	25-30 ft (intermediate)
PER-3	4/10/2002	Downgradient	9 ft	2-9 ft (shallow)
PER-3D	7/19/2013	Downgradient	30 ft	25-30 ft (intermediate)
PER-9	9/11/2013	Side-gradient, Downgradient	15 ft	1-15 ft (shallow)
PER-9D	9/11/2013	Side-gradient, Downgradient	35 ft	28-35 ft (intermediate)
PER-9DD	9/11/2013	Side-gradient, Downgradient	65 ft	55-65 ft (deep)
PER-10	7/16/2013	Downgradient	15 ft	3-15 ft (shallow)
PER-10D	7/19/2013	Downgradient	30 ft	25-30 ft (intermediate)
AB-4D	7/19/2013	Downgradient	30 ft	25-30 ft (intermediate)
AB-4R	6/23/2014	Downgradient	12.5 ft	2.5-12.5 ft (shallow)
AB-5	4/12/2002	Downgradient	10 ft	2-10 ft (shallow)

*Only monitoring wells that are representative of groundwater conditions associated with AOC 12 have been included in this table. Several proximal monitoring wells have been excluded since they are monitoring groundwater impacts associated with other AOCs at the Site.

4.1 2002-2014 Groundwater Analytical Results Summary

An evaluation of groundwater concentrations has been conducted for the previous three years of groundwater monitoring. The following provides a brief summary of historic groundwater analytical exceedances since 2013 for the monitoring wells associated with AOC 12. A summary of historic groundwater analytical results is provided as **Table 1**.

Volatile Organic Compounds (VOCs)

Several VOCs have been historically detected in the monitoring wells associated with AOC 12. These VOCs include benzene, bromodichloromethane, dibromodichloromethane, 1,2-dibromoethane, MTBE, TBA, and TCE. The following tables summarize GWQS exceedances only.

Sampling Date – July 2013

Sample ID	GWQS (ug/L)	PER-2D	PER-3D	PER-10	PER-10D	AB-4D
Benzene	1	ND	ND	**1.7 J**	**2.7**	0.33 J
Bromodichloromethane	1	**4.9**	0.88 J	ND	**2.3**	**6.9**
Dibromochloromethane	1	0.94 J	ND	ND	0.39 J	**1.5**
Methyl Tert Butyl Ether (MTBE)	70	ND	**195**	5.9	6.9	0.94 J
Trichloroethene	1	0.80 J	**3.1**	ND	8.9	**1.8**

Sampling Date – December 2013

Sample ID	GWQS (ug/L)	PER-2D	PER-9D	PER-9DD	PER-10D	AB-4D
Benzene	1	ND	ND	ND	**4.8**	ND
Bromodichloromethane	1	**1.6**	**4**	**3.7**	ND	**5.8**
Dibromochloromethane	1	0.45 J	0.71 J	0.68	ND	**1.5**
1,2-Dibromoethane	0.03	ND	**0.63 J**	**1.1**	ND	ND
Trichloroethene	1	ND	ND	ND	0.74 J	**2.8**

Sampling Date – November 2014

Sample ID	GWQS (ug/L)	PER-9DD	PER-10D
Benzene	1	ND	**17.2**
Bromodichloromethane	1	**2**	ND
1,2-Dibromoethane	0.03	**0.46 J**	ND
Tert Butyl Alcohol	100	ND	**194**

SVOCs

The only two (2) SVOC compounds historically detected at levels exceeding the GWQS are bis(2-Ethylhexyl)phthalate and benzo(a)anthracene. Bis(2-Ethylhexy)phthalate was detected at a maximum concentration of 94 ppb in 2009, exceeding the GWQS of 3 ppb. This compound was not detected over the GWQS again until 2015 (discussed below). Benzo(a)anthracene was detected at a maximum concentration of 0.3 ppb in 2010, exceeding the GWQS of 0.1 ppb. This compound was not detected above GWQS in subsequent sampling rounds.

Metals

Several metals including aluminum, arsenic, iron, manganese, silver, sodium, zinc, vanadium, beryllium, cadmium, and lead exceeded the GWQS for multiple sampling events. However, the GWQS for aluminum, iron, manganese, silver, and sodium are not health based standards and are based on secondary characteristics. Therefore, these metals are not considered contaminants of concern for

AOC 12. The metals which are considered contaminants of concern for AOC 12 include arsenic, lead, and chromium. Arsenic and lead impacts in the groundwater are potentially due to the presence of historic fill on the Site. Chromium has not been historically detected at levels exceeding the GWQS in AOC 12 wells.

	Compounds	Arsenic	Lead
	GWQS	3	5
PER-2	5/13/2002	**29.9**	**51.4**
PER-2	9/9/2010	**18**	**65**
PER-2	11/26/2012	**<60**	**<60**
PER-2	11/11/2013	**5.2**	**16.4**
PER-2	11/18/2014	**3.4**	**9.1**
PER-3	5/13/2002	**13.6**	**12.2**
PER-3	9/2/2009	**8.9**	<3
PER-3	9/9/2010	**9.3**	4.9
PER-3	11/26/2012	**<30**	**<30**
PER-3	11/11/2013	**5.8**	<5
PER-3	11/18/2014	<3	**5.1**
PER-3D	7/25/2013	**4.5**	3
PER-3D	11/18/2014	**3.1**	<3
PER-9	11/11/2013	**120**	**122**
PER-9	11/18/2014	**3.2**	4.2
PER-9D	11/18/2014	**3.4**	<3
PER-10	7/25/2013	**16.6**	**6.8**
PER-10	11/11/2013	**20.3**	<5
PER-10D	7/25/2013	**29.3**	<3
PER-10D	12/4/2013	**4.5**	<5
PER-10D	11/18/2014	**6.3**	<3
AB-4D	7/25/2013	**4.1**	<3.0
AB-4D	12/4/2013	**3.7[g]**	<5.0[g]
AB-4D	11/14/2014	**7.2**	<3.0
AB-4	5/13/02	**17**	**10.2**
AB-4	09/01/09	<3.0	**8.3**
AB-4	11/14/14	**18.3**	<9.0[c]
AB-5	5/13/02	**10.4**	4.9
AB-5	09/08/10	**8.2**	**6**

4.2 2015 Groundwater Analytical Results Summary

The following tables summarize the 2015 groundwater sampling results for AOC 12.

Volatile Organic Compounds (VOCs)

During the 2015 groundwater sampling event(s), no VOC exceedances were detected in groundwater samples collected from wells AB-4D, PER-1, PER-10, PER-2, PER-3, PER-9, PER-9D. The only VOCs detected over the standard were benzene, bromodichloromethane, 1,2-dibromoethane, MTBE, and TBA.

Client Sample ID:		NJ GWQS	PER-2D	PER-3D	PER-9DD	PER-10D
Date Sampled:			11/20/2015	11/23/2015	11/23/2015	11/23/2015
GC/MS Volatiles (SW846 8260C)						
Benzene	ug/l	1	ND (0.24)	ND (0.24)	ND (0.24)	**2**
Bromodichloromethane	ug/l	1	ND (0.23)	ND (0.23)	**1.5**	ND (0.23)
1,2-Dibromoethane	ug/l	0.03	ND (0.23)	ND (0.23)	**0.25 J**	ND (0.23)
Methyl Tert Butyl Ether	ug/l	70	**693**	**77**	0.31 J	25.7
Tert Butyl Alcohol	ug/l	100	**204**	45.8	ND (2.8)	35

Based on a review of groundwater analytical results, the predominant groundwater impacts for AOC 12 are VOC compounds present in the intermediate groundwater zone.

Semi-Volatile Compounds (SVOCs)

The only SVOCs detected over the GWQS during the 2015 sampling event were 1,4-dioxane and bis(2-Ethylhexyl)phthalate.

Client Sample ID:		NJ GWQS	AB-4D	PER-9D	PER-10D
Date Sampled:			11/23/2015	11/23/2015	11/23/2015
GC/MS Semi-volatiles (SW846 8270D)					
1,4-Dioxane	ug/l	0.4	**3.4**	ND (0.72)	**4.3**
bis(2-Ethylhexyl)phthalate	ug/l	3	ND (0.55)	**3.5**	ND (0.55)

Metals and Ammonia

Metals were detected in several wells during the 2015 groundwater sampling event. However, as discussed, metals for which groundwater standards are not health based are not being evaluated as Site contaminants of concern. Therefore, lead, arsenic, and chromium are the only metals being evaluated.

Ammonia exceeded the standard of 3 ppb in the samples collected from wells AB-4D, PER-3D, and PER-10D, with the highest concentration detected in well PER10D at 18.7 ppb.

Client Sample ID:		NJ GWQS	AB-4D	PER-2	PER-3D	PER-9	PER-10	PER-10D
Date Sampled:			11/23/2015	11/20/2015	11/23/2015	11/23/2015	11/23/2015	11/23/2015
Metals Analysis								
Arsenic	ug/l	3	**13.5**	<3.0	<3.0	**5.6**	**4.7**	<3.0
Chromium	ug/l	70	<10	<10	<10	<10	<10	<10
Lead	ug/l	5	<30 [i]	**5.5**	<3.0	3.3	<3.0	<3.0
General Chemistry								
Nitrogen, Ammonia	mg/l	3	**6.1**	0.56	**8.1**	<0.20	0.94	**18.7**

Low level metal impacts have been detected in both shallow and intermediate groundwater monitoring wells. Based on the groundwater analytical results, the contaminants of concern for AOC 12 are arsenic and lead. Both compounds are potentially related to the placement of historic fill across the Site.

A summary of 2015 groundwater analytical results is included on **Table 2** and depicted on **Figure 2**.

5.0 REMEDIAL INVESTIGATION WORKPLAN

The Site detention basin and off-site portions of Smith Creek have been potentially impacted due to former releases, the presence of historic fill, and historic operations on the Site. Contamination migration pathways include overland flow, direct discharges, and groundwater migration. Therefore, sediment and surface water sampling are required to assess and delineate any impacts.

Based on groundwater monitoring results, the contaminants of concern include benzene, MTBE, TBA, arsenic, lead, and ammonia. The arsenic and lead groundwater impacts are likely due to the presence of historic fill across the Site.

In order to assess impacts to the Site detention basin, the following remedial investigation activities are recommended for the on-site portions of AOC 12:

- Altered frequency of groundwater sampling for AOC 12 wells from annually to quarterly;
- Obtaining a Letter of Interpretation (LOI) from the NJDEP to confirm delineation and classification of phragmites dominated wetlands adjacent to the detention basin;
- Surface water and sediment sampling for the Site detention basin;

<u>Off-Site Sampling of Smith Creek</u>

As per 7:26E-3.6 in the TRSR, if there is the possibility that surface water and/or sediment has been impacted, then surface water and sediment samples must be collected to evaluate for the presence and level of contamination. Historically, there was a known release of eight (8) million gallons of oil on the Site. The majority of the crude oil was contained within a bermed area on the Site, but an undetermined amount of oil did flow into Smith Creek. In addition to the known release to Smith Creek, there is also the potential that contamination historically migrated off-site via overland flow and/or through the migration of impacted groundwater.

Furthermore, the NJDEP issued a Notice of Deficiency (NOD) on May 21, 2008 regarding the September 28, 2007 RIW. The RIW proposed sampling in the detention basin only. The NJDEP stated that the RIW failed to delineate horizontal and vertical limits of contamination in all media and that the "portion of Smith Creek that extends from the refinery to the Arthur Kill must also be included in the workplan."

Therefore, based on the applicable regulations and the requirements of the 2008 NJDEP NOD, the following surface water and sediment sampling plan is proposed for the off-site Smith Creek and adjacent pond. The sampling plan will include sampling background portions of the stream or other streams in the area to provide background data that can be evaluated as part of the surface water and sediment sampling proposed.

5.1 Groundwater Monitoring

All groundwater samples will be collected in accordance with the NJDEP *Field Sampling Procedures Manual* (FSPM). Prior to sampling, all monitoring wells will be gauged to determine the depth to water. A pneumatic power bladder pump will be used for purging at a slow rate following low flow techniques cited in Section 6.9.2.2 of the FSPM. The general chemistry of the purged ground water will be monitored to assess the quality of the recovered water. Once groundwater parameters are stable, a groundwater sample will be collected. Groundwater samples will be analyzed for Target Compound List (TCL) VOCs plus a forward library search, TCL SVOCs plus a forward library search, Target Analyte List (TAL) metals, and ammonia. In addition to the TCL VOC list, groundwater samples will also be analyzed for TBA and MTBE.

5.1.1 Quarterly Groundwater Monitoring

The monitoring wells associated with AOC 12 are currently sampled on an annual basis. In order to assess the possible seasonal impacts on groundwater contaminant concentrations, Earth Systems recommends collecting groundwater samples on a quarterly basis for the first year. Based on these results, the groundwater sampling frequency may be reduced. Once four (4) rounds of groundwater sampling are conducted, the analytical data will be evaluated. Groundwater samples will no longer be collected and analyzed for any monitoring wells that do not exhibit any impacts. Monitoring wells that have compounds in excess of the GWQS will continue to be sampled. Based on the quarterly groundwater results, additional wells may be installed as needed to complete horizontal and vertical delineation of groundwater impacts. For example, benzene was detected at 2 ppb in well PER-10D during the 2015 sampling event. After four rounds of sampling, if benzene is consistently detected over the standard then additional delineation will be necessary.

In addition to assessing possible seasonal effects on groundwater impacts, the goal of the additional rounds of sampling is to collect sufficient data to establish a Classification Exception Area (CEA) for AOC 12. A CEA requires a minimum of eight (8) rounds of analytical results and either demonstration that the groundwater contamination is naturally attenuating and exhibiting a downward trend or that the groundwater plume is stable.

5.2 Letter of Interpretation

According to NJDEP's Geoweb, adjacent to the detention basin are phragmites dominated and herbaceous wetlands. The wetlands are considered a potential suitable habitat for the following state threatened species: little blue heron, black crowned night heron, glossy ibis, and snowy egret. Site wetlands will be delineated and a request for a Letter of Interpretation (LOI) will be submitted to the NJDEP to confirm the delineation and to classify the wetlands. No invasive activities will be conducted within the wetlands prior to confirmation of the wetlands classification and determination if any permits are required.

5.3 Sediment and Surface Water Sampling

All sediment samples and surface water samples will be collected in accordance with the FSPM and applicable guidance. Sample collection will begin downstream and proceed to the upstream sample locations in order to minimize disturbance and to ensure that the samples are representative. All sediment and surface water samples will be collected at the same location; the surface water sample will be collected prior to collection of the sediment sample. The type of equipment utilized for collecting the sediment sampling will depend on the location of the sample. Sampling equipment will be selected accordingly in order to achieve the data quality objectives and to ensure the health and safety of sampling personnel.

Surface water samples will be collected from the detention basin, pond, and Smith Creek at a frequency described below. Prior to sampling; the size, depth, and flow of the water body will be recorded in a dedicated field book. In addition, the current tidal stage of the adjacent Arthur Kill will be recorded. A calibrated water quality meter will be utilized to record pH, temperature, conductivity, salinity, dissolved oxygen, ORP, and turbidity for all surface water samples.

Depending on access and the location of the proposed sampling locations, surface water samples will either be collected as a direct grab sample or with a depth sampler, such as a Kemmerer depth sampler. For grab samples, a sample container will be placed just below the surface of the water and allowed to fill to the required sampling volume. If necessary, surface water sampling equipment may be used for

grab sampling, such as a pond sampler. If a water sample is required at depth, the appropriate sampling device will be used to isolate the desired interval and the depth of the sample will be recorded in the field book. If sampling equipment is used, it will be decontaminated in accordance with the Quality Assurance Project Plan (**Appendix 1**) between sampling locations. Surface water samples collected using sampling equipment will be transferred directly into the laboratory provided glassware. All surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride. Analytical results will be compared initially to the applicable, most conservative ecological screening level.

Sediment samples will be collected at a frequency described below. Sediment is a heterogeneous material and the correct sampling equipment and collection methods must be utilized to ensure that a representative sample is collected. Care must be taken to minimize disturbance and the loss of fines as the sample is retrieved through the water column. Any water that is collected along with the sediment sample will be transferred to the laboratory provided jar and will not be decanted off the sample. Sediment samples will be biased towards observed depositional areas and collected using a sediment coring device or dredge. A sample will be collected in the 0 to 6-inch surficial interval as well as from the 6 to 12-inch interval below grade. Observations regarding sediment color, particle size, and any evidence of impacts will be recorded in a dedicated field book. All sediment samples will be analyzed for TCL VO+15, TCL SVOCs+15, TAL metals, Total Organic Carbon (TOC) pH, Extractable Petroleum Hydrocarbons (EPH) and grain size. Analytical results will be compared initially to the applicable, most conservative ecological screening level.

5.3.1 Detention Basin Surface Water/Sediment Sampling

The detention basin has been historically pumped out and the water discharged to the water treatment plant. The detention basin sediments and surface water could potentially be impacted with Site contaminants of concern due to historic releases, overland flow, direct discharges to the basin, and the migration of contaminated groundwater. A total of twelve (12) locations are proposed for the collection of sediment and surface water samples. Eight (8) co-located sediment and surface water samples will be collected along the perimeter of the basin and biased towards any inflows, outflows, or areas of distressed vegetation. As described in **Section 5.3**, sediment samples will be collected from both the surface (0 to 6-inch interval) as well as from 6 to 12 inches below grade.

In addition, four (4) surface water and sediment samples will be collected from the interior portion of the basin. A water level gauge is present in the detention basin. The depth of the basin will be recorded at the time of sampling and a calibrated water meter will be lowered in the water to determine if there are distinct differences in temperature at different depths. Depending on observed conditions, surface water samples may be collected at multiple depths. If surface water conditions appear uniform, the sample will be collected from either just below the surface or at mid-depth. Sediment samples will be collected utilizing a dredge sampler or coring device. An attempt will be made to collect samples from both 0 to 6 inches and 6 to 12 inches as discussed in **Section 5.3**. However, collecting a sample below the top 6 inches of sediment may be difficult due to the depth of the basin at the time of sampling.

As described above, surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride. Sediment samples will be analyzed for TCL VO+15, TCL SVOCs+15, TAL metals, TOC, pH, EPH, and grain size.

Proposed detention basin sampling locations are depicted on **Figure 3.**

5.3.2 Smith Creek Surface Water/Sediment Sampling (Off-Site)

The first step in sampling Smith Creek and the associated pond is to arrange access through the current owners of the property. As described below, the pond and first section of Smith Creek are located on property owned by PSE&G. The remaining sections of Smith Creek can be accessed through a public park owned by Woodbridge Township or through multiple commercial and residential property owners. In order to simplify coordinating access, access will only be arranged through PSE&G and Woodbridge Township. Smith Creek will not be accessed from the west bank through the residential or commercial properties.

For sampling purposes, the Creek has been divided into sections. Sediment and surface water sampling events will be conducted in iterative phases. Analytical data will be reviewed and a determination will be made whether additional sampling is required after the initial phase is completed. In addition to the Creek, there is a pond area that does not appear directly connected to Smith Creek. Since the pond is also downgradient from the Site and is in the approximate same location of the historic Smith Creek channel, the pond will be sampled as well, as discussed below. **Figure 3** illustrates the proposed locations of all Smith Creek and pond sampling locations.

5.4 Phase I Smith Creek Surface/Sediment Water Sampling

<u>Block 756, Lot 2, Block 760 Lot 1.01 (Owner – PSE&G)</u>

<u>Pond (10,000 square feet)</u>

The pond is approximately 10,000 square feet and is currently located to the north of Smith Creek. A thorough visual inspection will be conducted prior to conducting sampling. It is unclear whether PSE&G routes any kind of discharge to the pond. In the process of arranging access to the pond with PSE&G, Earth Systems will inquire about any inflows or outflows associated with the pond. Due to the presence of large stone surrounding the pond and possibly at the base of the pond, it is anticipated that only surface water samples will be collected. However, if sediment is present in the pond, sediment samples will be collected as well. Samples will be collected from two (2) locations.

As described above, surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride.

The locations of the surface water samples are depicted on **Figure 3**.

<u>Creek Section 1 (~900 feet)</u>

Sediment and surface water samples will be collected from nine (9) locations, approximately one (1) sample per every 300 linear feet, as depicted on **Figure 3**. Three (3) sediment and surface water samples will be collected from the north bank and three (3) sediment and surface water samples will be collected from the south bank. Sediment samples will be collected from both 0 to 6 inches and 6 to 12 inches below grade. Three (3) sediment and surface water samples will also be collected from the approximate center of the Creek channel. Sediment samples collected in the center of the Creek will only be collected from surficial sediment (0 to 6 inches).

As described above, surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride. Sediment samples will be analyzed for TCL VO+15, TCL SVOCs+15, TAL metals, TOC pH, EPH, and grain size.

The locations of Section 1 Creek sampling are depicted on **Figure 3**.

<u>Block 756, Lot 1.01 (Owner – Woodbridge Township) & Block 760, Lot 1 (County of Middlesex Treasurer)</u>

<u>Creek Section 2 (~600 Feet)</u>
Sediment and surface water samples will be collected from six (6) locations, two (2) each from the north and south banks, as well as two from the approximate center of the Smith Creek channel. Sediment samples collected from the Creek banks will be collected from both 0 to 6 inches and 6 to 12 inches below grade. Sediment samples collected in the center of the Creek will only be collected from surficial sediment (0 to 6 inches).

The locations of Section 2 Creek sampling are depicted on **Figure 3**.

<u>Creek Section 3 – Creek changes direction to the west (~700 feet)</u>
The third section of Smith Creek meanders towards the west. Based on the flow direction of the Creek, and that the channel direction changes in this section, the southern bank is possibly a depositional area. Sediment and surface water samples will be collected from two (2) locations along the southern bank. A marina is located adjacent to the northern bank, therefore access to this area may be limited. If accessible, sediment and surface water samples will be collected from two (2) locations along the northern bank. Two (2) surface water and sediment samples will also be collected from the approximate center of this Creek section. Sediment samples collected from the Creek banks will be collected from both 0 to 6 inches and 6 to 12 inches below grade. Sediment samples collected in the approximate center of the Creek will only be collected from surficial sediment (0 to 6 inches).

As described above, surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride. Sediment samples will be analyzed for TCL VO+15, TCL SVOCs+15, TAL metals, TOC, pH, EPH, and grain size.

The locations of Section 3 Creek sampling are depicted on **Figure 3**.

5.5 Phase II Smith Creek Surface Water/Sediment Sampling

Due to potential access issues, as well as the width and potential significant depth of this section of Smith Creek, sediment and surface water samples will initially be collected from Smith Creek sections 1, 2, and 3, as described above and depicted on **Figure 3**. After the analytical results are tabulated and interpreted from the Phase I sampling event, a determination will be made whether further downgradient sampling is necessary. If delineation is necessary, sampling will be conducted for the 4th section of Smith Creek (as depicted on **Figure 3**).

5.6 Analytical Data Review and Evaluation

At the completion of sample collection for the detention basin and Smith Creek sections 1, 2, and 3; sediment and surface water sample analytical results will be tabulated, interpreted, and compared to the most conservative applicable ecological screening levels. If the analytical results are below the ecological screening levels, no additional investigation or remediation will be required.

If the analytical results exceed the most conservative ecological screening levels, the results will be evaluated in conjunction with several additional factors such as potential additional sources of impacts, as well as comparison and evaluation of the data against less conservative ecological screening levels, if applicable. Based on this evaluation, a determination will be made if additional investigation is

warranted for horizontal and vertical delineation, to represent a different tidal stage, or for ecological toxicology assessment purposes.

In addition, since there is no upgradient portion of Smith Creek, the surrounding area will be evaluated to determine if there is a stream that could be sampled as a reference stream to assess background levels indicative to the area.

Analytical data obtained during the RIW for Smith Creek will be incorporated into the overall site-wide ecological assessment, as well as the EPA 725 and 750 Environmental Indicator Reports.

5.7 Quality Assurance Project Plan

Samples will be collected in accordance with the sampling procedures outlined in the Quality Assurance Project Plan (QAPP), which is included as **Appendix 1**. The QAPP will provide guidance to the project team to ensure all field activities are completed in a manner consistent with the NJDEP requirements and that all data produced is of sufficient quality to meet NJDEP standards. Analytical data packages will be presented in the New Jersey Reduced Deliverables format, including electronic data deliverables (EDDs).

5.8 Health and Safety Plan

Sediment and surface water sampling present unique challenges. The Health and Safety Plan (HASP) will address the specific issues associated with this kind of sampling and the safety of all personnel will be the first priority. A Site specific HASP will be prepared in accordance with NJAC 7:26E-1.9. All Site personnel will be informed prior to performing any site activities of all health and safety protocols.

6.0 RECEPTOR EVALUATION

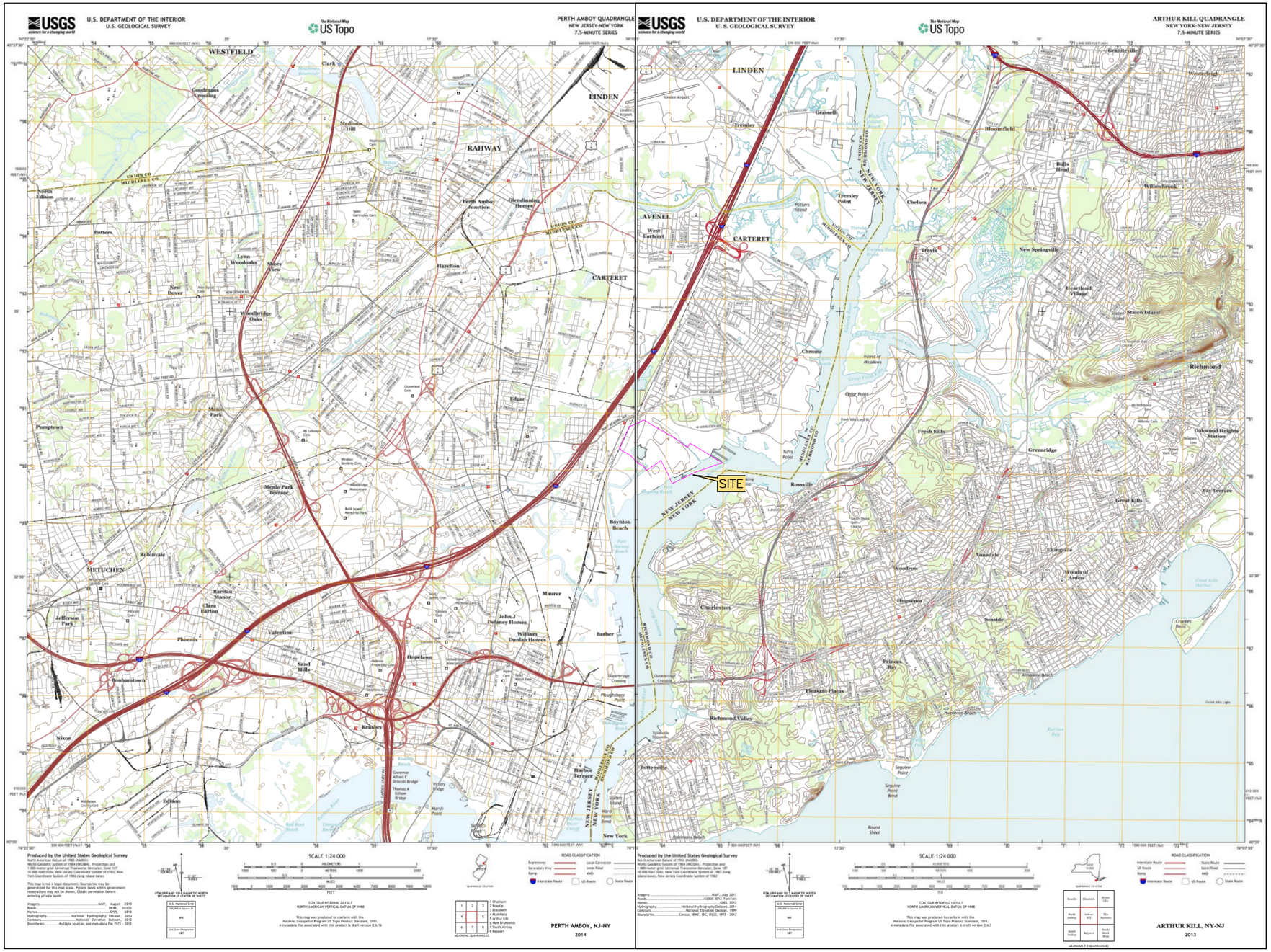
Pursuant to N.J.A.C. 7:1E-1.12 through 1.16 of the NJDEP TRSR, a Receptor Evaluation (RE) was prepared for the Site and submitted with the Site Investigation Report submitted in November 2015.

Pursuant to Chapter 7:26E-1.16 of the NJDEP TRSR, an Ecological Evaluation (EE) was conducted as part of the SI activities. An EE is a screening-level ecological risk assessment that serves to determine whether more rigorous ecological risk evaluations are warranted, and if so, to narrow the scope of subsequent activities. The EE conducted during 2015 SI activities concluded that an Ecological Risk Assessment (ERA) was required. Analytical data collected during the AOC 12 remedial investigation activities detailed in this plan will be incorporated into the ERA being prepared for the Site. The ERA will determine whether actual or potential ecological risks exist at the site and generate data for risk-based remediation goal determinations and for any Risk Management Decisions (RMD).

7.0 SCHEDULE

This RIW proposes remedial investigation activities relating to AOC 12: Smith Creek and Detention Basin. In accordance with the NJDEP's Technical Requirements for Site Remediation, HC-PR will provide the NJDEP with 14 days' notice for all field activities prior to commencement of work. If warranted, the RIR will include recommendations for additional investigation activities.

FIGURES



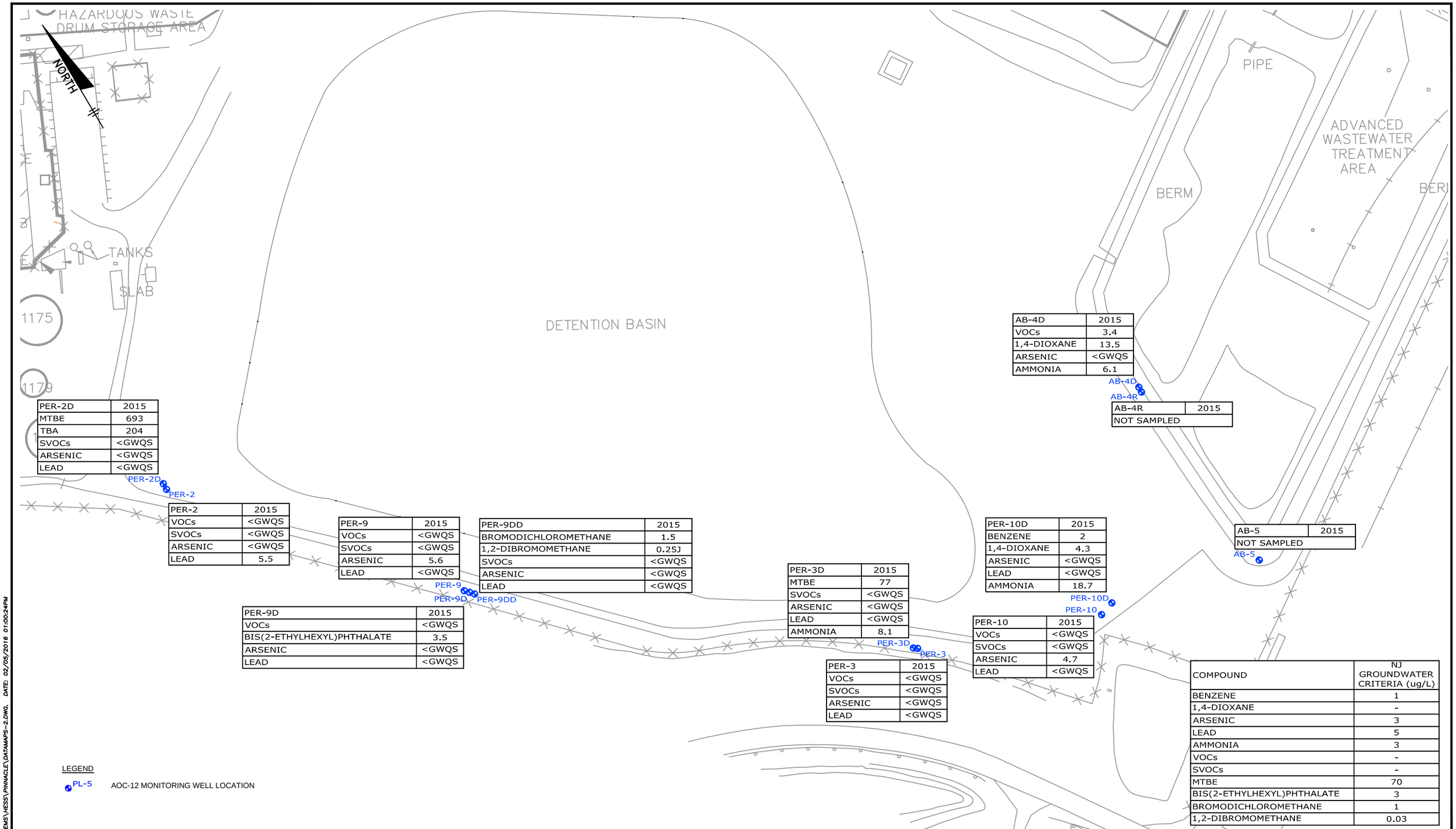
USGS MAP

Hess Corporation Former Port Reading Complex (HC–PR)
750 Cliff Road
Port Reading, New Jersey

Earth Systems

Figure 1

09/15 FIG1



PER-2D	2015
MTBE	693
TBA	204
SVOCs	<GWQS
ARSENIC	<GWQS
LEAD	<GWQS

PER-2	2015
VOCs	<GWQS
SVOCs	<GWQS
ARSENIC	<GWQS
LEAD	5.5

PER-9	2015
VOCs	<GWQS
SVOCs	<GWQS
ARSENIC	5.6
LEAD	<GWQS

PER-9D	2015
VOCs	<GWQS
BIS(2-ETHYLHEXYL)PHTHALATE	3.5
ARSENIC	<GWQS
LEAD	<GWQS

PER-9DD	2015
BROMODICHLOROMETHANE	1.5
1,2-DIBROMOMETHANE	0.25J
SVOCs	<GWQS
ARSENIC	<GWQS
LEAD	<GWQS

PER-3D	2015
MTBE	77
SVOCs	<GWQS
ARSENIC	<GWQS
LEAD	<GWQS
AMMONIA	8.1

PER-3	2015
VOCs	<GWQS
SVOCs	<GWQS
ARSENIC	<GWQS
LEAD	<GWQS

PER-10D	2015
BENZENE	2
1,4-DIOXANE	4.3
ARSENIC	<GWQS
LEAD	<GWQS
AMMONIA	18.7

PER-10	2015
VOCs	<GWQS
SVOCs	<GWQS
ARSENIC	4.7
LEAD	<GWQS

AB-4D	2015
VOCs	3.4
1,4-DIOXANE	13.5
ARSENIC	<GWQS
AMMONIA	6.1

AB-4R	2015
NOT SAMPLED	

AB-5	2015
NOT SAMPLED	

COMPOUND	NJ GROUNDWATER CRITERIA (ug/L)
BENZENE	1
1,4-DIOXANE	-
ARSENIC	3
LEAD	5
AMMONIA	3
VOCs	-
SVOCs	-
MTBE	70
BIS(2-ETHYLHEXYL)PHTHALATE	3
BROMODICHLOROMETHANE	1
1,2-DIBROMOMETHANE	0.03

LEGEND

PL-5 AOC-12 MONITORING WELL LOCATION

Earth Systems

SCALE: 0 100 SCALE: 1"=100'

DATE: 2/5/16
DRAWN BY: AG
CHECKED BY: JC
FILENAME: DATAMAPS-2
LAYOUT: 1

PROJECT:
HESS CORPORATION
FORMER PORT READING COMPLEX (HC-PR)
750 CLIFF ROAD
PORT READING, NEW JERSEY

TITLE:
AOC 12: 2015 GROUNDWATER ANALYTICAL RESULTS

FIGURE NO.
2

FILE: F:\CLIENTS\E\EARTHSYSTEMS\HESS\PINNACLE\DATAMAPS-2.DWG, DATE: 02/05/2016 01:00:24PM

NORTH

LEGEND

- PROPERTY BOUNDARY
- SURFACE WATER SAMPLE
- SURFACE WATER AND SEDIMENT SAMPLE
- HS — HISTORIC SPILL LOCATIONS

Earth Systems

SCALE: 0 — 600 SCALE: 1"=600'

DATE: 2/5/16
DRAWN BY: AG
CHECKED BY: JC
FILENAME: SED_SAMPLES
LAYOUT: SITE PLAN

PROJECT:

HESS CORPORATION

FORMER PORT READING COMPLEX (HC-PR)
750 CLIFF ROAD
PORT READING, NEW JERSEY

TITLE:

AOC 12: SEDIMENT AND SURFACE WATER SAMPLE MAP

FIGURE NO.

3

FILE: I:\CLIENTS\E\EARTHSYSTEMS\HESS\PINNACLE\SED_SAMPLES.DWG DATE: 08/17/2016 02:37:22PM

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TABLES

Table 1
Hess Corporation Former Port Reading Complex (HC-PR)
AOC 12: Smith Creek and Detention Basin
750 Cliff Road
Port Reading, Middlesex County, New Jersey

Summary of Historic Groundwater Analytical Results

Sample ID	Groundwater Quality Standards	PER-2	PER-2	PER-2	PER-2	PER-2	PER-2	PER-2	PER-2D	PER-2D	PER-2D	PER-3	PER-3	PER-3	PER-3	PER-3	PER-3	PER-3
Sample Date		5/13/2002	9/2/2009	9/9/2010	9/20/2011	11/26/2012	11/11/2013	11/18/2014	7/25/2013	12/4/2013	11/18/2014	5/13/2002	9/2/2009	9/9/2010	9/22/2011	11/26/2012	11/11/2013	11/18/2014
Matrix		GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW
Units		ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
Metals																		
Aluminum	200	**17100**	**509**	**15000**	NA	**329**	**4250**	**775**	**871**	<200	<200	**2360**	**238**	**1630**	NA	**7360**	**1540**	**1470**
Antimony	6	<5	<6	<6	NA	<6	<6	<6	<6	<6	<6	<5	<6	<6	NA	<6	<6	<6
Arsenic	3	**29.9**	<3	**18**	NA	**<60**	**5.2**	**3.4**	<3	<3	<3	**13.6**	**8.9**	**9.3**	NA	**<30**	**5.8**	<3
Barium	6000	<200	<200	<200	NA	485	70	<200	<200	66.2	<200	<200	<200	<200	NA	231	122	<200
Beryllium	1	NA	<	**3.9**	NA	<1	**<4**	<1	<1	**<4**	<1	NA	<1	<1	NA	1	**<4**	<1
Cadmium	4	NA	<3	<3	NA	<3	<4	<3	<3	<4	<3	NA	<3	<3	NA	**8.2**	<4	<3
Calcium		24400	22800	22800	NA	547000	57000	217000	26100	57800	106000	40700	18000	102000	NA	371000	146000	9340
Chromium	70	31	<10	26	NA	<10	<10	<10	<10	<10	<10	<10	<10	<10	NA	<10	<10	<10
Cobalt	100	NA	<50	<50	NA	<50	<50	<50	<50	<50	<50	NA	<50	<50	NA	<50	<50	<50
Copper	1300	63.6	17.4	288	NA	18.5	83.3	30.7	<10	<25	<10	<25	<10	36.3	NA	41.4	<25	13.1
Iron	300	**30700**	**3240**	**16800**	NA	**9320**	**15700**	**1960**	**2180**	<100	106	**54000**	**7940**	**11200**	NA	**410**	**5600**	**2280**
Lead	5	**51.4**	3.1	**65**	NA	**<60**	**16.4**	**9.1**	3.3	<5	<3	**12.2**	<3	4.9	NA	**<30**	<5	**5.1**
Magnesium		5330	<5000	<5000	NA	158000	12300	<5000	7800	12900	19100	60100	9010	65300	NA	321000	259000	15200
Manganese	50	**263**	43	**175**	NA	**708**	**922**	**105**	**114**	<15	<15	**513**	**535**	**1670**	NA	**1860**	**1590**	26.9
Mercury	2	<0.2	<0.2	0.41	NA	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.25	<0.2	0.29	NA	<0.2	<0.2	<0.2
Nickel	100	NA	<10	45.6	NA	**158**	<40	<10	<10	<40	<10	NA	<10	<10	NA	<100	<40	<10
Potassium		21100	<10000	<10000	NA	27800	6360	<10000	<10000	<5000	<10000	32000	<10000	15600	NA	84400	77400	<10000
Selenium	40	5	<10	<10	NA	<10	<10	<10	<10	<10	<10	5	<10	<10	NA	<10	<10	<10
Silver	40	NA	<10	<10	NA	<10	<5	<10	<10	<5	<10	NA	<10	<10	NA	<10	<5	<10
Sodium	50000	22700	<10000	<10000	NA	**851000**	**141000**	42500	28700	38700	48500	**599000**	**104000**	**596000**	NA	**2770000**	**2680000**	**167000**
Thallium	2	NA	<2	<2	NA	<2	<2	<2	<2	<2	<2	NA	<2	<2	NA	**<20**	<2	<2
Vanadium	60	**100**	<50	<50	NA	<50	14.9	<50	<50	<10	<50	<50	<50	<50	NA	<50	<10	<50
Zinc	2000	101	22.5	735	NA	41	278	69.7	20.6	<20	<20	40.2	<20	29.4	NA	235	22.8	<20
Volatile Organic Compounds																		
Acetone	6000	ND	9 J	ND	15.3	ND	ND	7.6 J	ND	ND	ND	4.1	ND	ND	ND	ND	ND	ND
Benzene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bromodichloromethane	1	NA	ND	ND	ND	ND	ND	ND	**4.9**	**1.6**	ND	NA	ND	ND	ND	ND	ND	ND
2-Butanone (MEK)	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Carbon disulfide	700	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Carbon Tetrachloride	1	NR	NR	NR	NR	NR	ND	ND	NR	ND	ND	NR	NR	NR	NR	NR	ND	ND
Chlorobenzene	50	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroethane	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	70	ND	ND	ND	ND	ND	ND	ND	15.4	3.6	ND	ND	ND	ND	ND	ND	ND	ND
Dibromochloromethane	1	NR	NR	NR	NR	NR	ND	ND	0.94 J	0.45 J	ND	NR	NR	NR	NR	NR	ND	ND
1,2-Dibromoethane	0.03	NR	NR	NR	NR	NR	ND	ND	NR	ND	ND	NR	NR	NR	NR	NR	ND	ND
1,2 - Dichlorobenzene	600	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,3 - Dichlororbenzene	600	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,4 - Dichlorobenzene	75	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Dichlorodiflurorometh ane	1000	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethane	50	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichloroethane	2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
cis - 1,2 - Dichloroethene	70	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
trans - 1,2 - Dichlororethene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichlororpropane	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Freon 113		NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
2-Hexanone	300	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Isopropylbenzene	700	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Methylcyclohexane		NA	ND	ND	ND	ND	0.47 J	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Methyl Tert Butyl Ether (MTBE)	70	NA	ND	ND	ND	ND	ND	ND	ND	1	1.4	NA	ND	ND	ND	ND	ND	ND
4-Methyl-2-pentanone (MIBK)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Methylene Chloride	3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	600	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3 - Trichlorobenzene		NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,2,4 - Trichlorobenzene	9	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,1,1 - Trichloroethane	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2 - Trichloroethane	3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	1	ND	ND	ND	ND	ND	ND	ND	0.80 J	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl Chloride	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes (Total)	1000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tert Butyl Alcohol	100	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	7.8 J	ND	ND	ND	ND
Total Tic, Volatile	100/500	490	0	0	0	0	0	0	0	0	0	1800	0	0	0	0	0	0
Semi-Volatile Organic Compounds																		
Acenaphthene	400	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Acenaphthylene	100	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Acetophenone	700	NA	ND	NA	NA	ND	ND	ND	ND	ND	ND	NA	ND	NA	NA	ND	ND	ND
Anthracene	2000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzaldehyde		NA	ND	NA	NA	ND	ND	ND	ND	ND	ND	NA	ND	NA	NA	ND	ND	ND
Benzo(a)anthracene	0.1	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(a)pyrene	0.1	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(b)fluoranthene	0.2	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(g,h,i)perylene	100	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(K)fluoranthene	0.5	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Butyl Benzyl phthalate	100	2.9	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.9	ND	ND	ND	ND	ND	ND
1,1-Biphenyl	400	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
4-Chloroaniline	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Carbazole		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Caprolactam	5000	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	4.1	ND	ND
Chrysene	5	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
3,3-Dichlorobenzidine	30	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
1,4-Dioxane	10	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Dibenzofuran		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-octyl phthalate	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Diethyl phthalate	6000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dimethyl phthalate	100	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
bis(2-ethylhex)phthalate	3	1.7	ND	**3.6**	**9.2**	2.1	ND	ND	ND	ND	ND	1.1	ND	**7.2**	ND	1.7 J	ND	ND
Dibenzo(a,h)anthracene	0.3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Fluoranthene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Fluorene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Hexachlorobenzene	0.02	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	0.2	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Isophorone	40	NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
2-Methylnaphthalene	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Naphthalene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Phenanthrene	100	ND	ND	ND	ND	ND	ND	ND	ND	0.135	ND	ND	ND	0.125	ND	ND	ND	ND
Pyrene	200	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4,5-Tetrachlorobenzene		NA	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND
Total Tic, Semi-Volatile	100/500	0	21.7 J	400 J	9.9 J	65.1 KJ	0	200.7 J	0	0	0	117.4	22 J	0	0	20 J	11 J	0

Table 1
Hess Corporation Former Port Reading Complex (HC-PR)
AOC 12: Smith Creek and Detention Basin
750 Cliff Road
Port Reading, Middlesex County, New Jersey

Summary of Historic Groundwater Analytical Results

Sample ID	Groundwater Quality Standards	PER-3D	PER-3D	PER-3D	PER-9	PER-9	PER-9D	PER-9D	PER-9DD	PER-9DD	PER-10	PER-10	PER-10	PER-10D	PER-10D	PER-10D	AB-4D	AB-4D
Sample Date		7/25/2013	12/4/2013	11/18/2014	11/11/2013	11/18/2014	12/4/2013	11/18/2014	12/4/2013	11/18/2014	7/25/2013	11/11/2013	11/18/2014	7/25/2013	12/4/2013	11/18/2014	7/25/2013	12/4/2013
Matrix		GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW
Units		ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
Metals																		
Aluminum	200	**308**	<200	<200	**73600**	**1470**	**376**	**394**	**368**	**239**	**334**	**401**	**376**	<200	**327**	<200	**377**	<200[g]
Antimony	6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6.0	<6.0[g]
Arsenic	3	**4.5**	<3	**3.1**	**120**	**3.2**	<3	**3.4**	<3	<3	**16.6**	**20.3**	<3	**29.3**	**4.5**	**6.3**	**4.1**	**3.7[g]**
Barium	6000	<200	<50	<200	321	<200	<50	<200	<50	<200	<200	339	<200	<200	132	1150	<200	<50[g]
Beryllium	1	<1	**<4**	<1	**<4**	<1	**<4**	<1	**<4**	<1	<1	**<4**	<1	<1	<1	<1	<1.0	<1.0[g]
Cadmium	4	<3	<4	<3	<4	<3	<4	<3	<4	4	<3	<4	<3	<3	<4	<3	<3.0	<4.0[g]
Calcium		33300	37900	55500	78300	16300	40400	71800	38300	40000	93300	225000	30000	41400	5000	159000	26,900	30,600[g]
Chromium	70	<10	<10	<10	206	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10[g]
Cobalt	100	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50[g]
Copper	1300	<10	<25	<10	291	22.1	<25	<10	<25	<10	<10	<25	12.6	<10	<25	<10	<10	<25[g]
Iron	300	**614**	**390**	<100	**206000**	**3350**	**469**	**850**	**1080**	**591**	**68400**	**342000**	**2180**	**1320**	**534**	<100	**459**	145[g]
Lead	5	3	<5	<3	**122**	4.2	<5	<3	<5	<3	**6.8**	<5	<3	<3	<5	<3	<3.0	<5.0[g]
Magnesium		70400	90300	105000	64600	9600	14600	29300	9150	10100	25200	123000	<5000	65200	<5000	6050	14,200	18,400[g]
Manganese	50	**150**	**167**	**143**	**1100**	**142**	15.3	<15	**62.4**	29.9	**1140**	**5610**	20.5	**160**	30.3	<15	44.6	<15[g]
Mercury	2	<0.2	<0.2	<0.2	1.4	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.20	<0.20[g]
Nickel	100	<10	<40	<10	88.2	<10	<40	<10	<40	<10	11.9	<40	<10	<10	53.5	60.8	<10	<40[g]
Potassium		53400	52300	57100	26200	<10000	6080	10100	<5000	<10000	30300	30300	<10000	68000	343000	118000	15,500	15,800[g]
Selenium	40	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10[g]
Silver	40	<10	<5	<10	<5	<10	<5	<10	<5	<10	<10	<5	<10	<10	<5	<10	<10	<5.0[g]
Sodium	50000	**604000**	**825000**	**889000**	**740000**	**148000**	**66500**	**159000**	**60300**	**76200**	**341000**	**1080000**	32000	**528000**	**764000**	**924000**	**105,000**	**142,000[g]**
Thallium	2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2.0	<2.0[g]
Vanadium	60	<50	<10	<50	**333**	<50	<10	<50	<10	<50	<50	<10	<50	<50	<10	<50	<50	18.3[g]
Zinc	2000	<20	<20	<20	276	<20	22.4	34.7	<20	43.1	<20	<20	<20	<20	<20	<20	<20	<20[g]
Volatile Organic Compounds																		
Acetone	6000	ND	ND	ND	ND	ND	ND	ND	ND	ND	30.3 J	ND	4.6 J	ND	47.1	49.5	ND	ND
Benzene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	**1.7 J**	**1.9**	ND	**2.7**	**4.8**	**17.2**	0.33 J	ND
Bromodichloromethane	1	0.88 J	ND	ND	ND	ND	**4**	0.93 J	**3.7**	**2**	ND	ND	ND	**2.3**	ND	ND	**6.9**	**5.8**
2-Butanone (MEK)	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	6.9 J	6.4 J	ND	ND
Carbon disulfide	700	1.3 J	ND	ND	0.47 J	ND	ND	ND	ND	ND	ND	ND	ND	1.9 J	1.3 J	ND	0.35 J	ND
Carbon Tetrachloride	1	NR	ND	ND	ND	ND	ND	ND	0.49 J	ND	ND	ND	ND	ND	ND	ND	NR	ND
Chlorobenzene	50	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.5	ND	ND
Chloroethane	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	70	3.3	ND	ND	ND	ND	12.6	2.6	13.6	6.3	ND	0.53 J	0.90 J	9.7	0.26 J	ND	23.8	18.6
Dibromochloromethane	1	ND	ND	ND	ND	ND	0.71 J	ND	0.68	0.36 J	ND	ND	ND	0.39 J	ND	ND	**1.5**	**1.5**
1,2-Dibromoethane	0.03	NR	ND	ND	ND	ND	**0.63 J**	ND	**1.1**	**0.46 J**	ND	ND	ND	ND	ND	ND	NR	ND
1,2 - Dichlorobenzene	600	ND	ND	ND	ND	ND	0.26 J	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,3 - Dichlororbenzene	600	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,4 - Dichlorobenzene	75	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dichlorodifluroromethane	1000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethane	50	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichloroethane	2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
cis - 1,2 - Dichloroethene	70	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
trans - 1,2 - Dichlororethene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichlororpropane	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.3 J	0.78 J	ND	2	1.5	3.5	ND	ND
Freon 113		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-Hexanone	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Isopropylbenzene	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.54 J	ND	0.47 J	0.33 J	0.38 J	ND	ND
Methylcyclohexane		ND	ND	ND	ND	ND	ND	ND	ND	ND	1.4 J	3.2 J	0.52 J	2.6 J	0.69 J	0.25 J	ND	ND
Methyl Tert Butyl Ether (MTBE)	70	**195**	169	0.65 J	ND	ND	5.1	0.98 J	ND	ND	5.9	ND	ND	6.9	3.4	3.4	0.94 J	ND
4-Methyl-2-pentanone (MIBK)		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	9.6	ND	ND	ND	ND
Methylene Chloride	3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	600	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.28 J	ND	0.32 J	ND	ND
1,2,3 - Trichlorobenzene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4 - Trichlorobenzene	9	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1 - Trichloroethane	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2 - Trichloroethane	3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	1	**3.1**	1.2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	8.9	0.74 J	ND	**1.8**	**2.8**
Vinyl Chloride	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes (Total)	1000	ND	ND	ND	ND	ND	ND	ND	ND	ND	7.2	1.9 J	ND	0.98 J	0.42 J	1.2 J	ND	ND
Tert Butyl Alcohol	100	30.5	30.4	ND	ND	ND	ND	ND	ND	ND	ND	32.9	ND	9.6 J	30.9	194	ND	ND
Total Tic, Volatile	100/500	0	6.6 J	0	0	0	0	0	0	0	0	0	0	0	5.1	12.6	0.0	0.0
Semi-Volatile Organic Compounds																		
Acenaphthene	400	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.16	0.484	ND	0.265	ND	ND	ND	ND
Acenaphthylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.283	ND	ND	ND
Acetophenone	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.5 J	1.7 J	ND	ND
Anthracene	2000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.112	ND	ND	ND
Benzaldehyde		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(a)anthracene	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	**0.14**	ND	**0.129**	**0.227**	ND	ND	ND
Benzo(a)pyrene	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(b)fluoranthene	0.2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(g,h,i)perylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzo(K)fluoranthene	0.5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Butyl Benzyl phthalate	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Biphenyl	400	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
4-Chloroaniline	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Carbazole		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Caprolactam	5000	ND	ND	ND	ND	ND	ND	ND	0.79 J	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chrysene	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3,3-Dichlorobenzidine	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,4-Dioxane	10	ND	ND	1.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dibenzofuran		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	700	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-octyl phthalate	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Diethyl phthalate	6000	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dimethyl phthalate	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
bis(2-ethylhex)phthalate	3	**5.6**	ND	ND	ND	ND	ND	3.1	ND	ND	ND	ND	ND	ND	ND	ND	1.0 J	ND
Dibenzo(a,h)anthracene	0.3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Fluoranthene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.225	ND	ND	0.115	0.158	ND	ND	ND
Fluorene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.9	0.831	ND	0.401	0.304	ND	ND	ND
Hexachlorobenzene	0.02	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	0.2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Isophorone	40	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.6 J	ND	ND	ND
2-Methylnaphthalene	30	ND	ND	ND	ND	ND	ND	ND	ND	ND	7.1	ND	ND	ND	2.7	3.2	ND	ND
Naphthalene	300	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.23	0.987	ND	ND	2.33	4.6	ND	ND
Phenanthrene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.57	0.522	ND	0.206	0.397	ND	ND	ND
Pyrene	200	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.134	ND	0.142	0.183	ND	ND	ND
1,2,4,5-Tetrachlorobenzene		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Total Tic, Semi-Volatile	100/500	18.5 J	0	12 J	0	0	0	0	0	0	810 J	374.1 J	161.6 J	37.4 J	149.1 J	1883 J	0.0	0.0

Table 1
Hess Corporation Former Port Reading Complex (HC-PR)
AOC 12: Smith Creek and Detention Basin
750 Cliff Road
Port Reading, Middlesex County, New Jersey

Summary of Historic Groundwater Analytical Results

Sample ID	Groundwater Quality Standards	AB-4D	AB-4	AB-4	AB-4	AB-4	AB-4	AB-4	AB-4	AB-5	AB-5	AB-5	AB-5	AB-5	AB-5	AB-5
Sample Date		11/14/2014	5/13/02	09/01/09	09/08/10	09/20/11	11/26/12	11/11/13	11/14/14	5/13/02	09/01/09	09/08/10	09/20/11	11/26/12	11/11/13	11/14/14
Matrix		GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW
Units		ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
Metals																
Aluminum	200	**246**	**7,360**	**5,200**	NS	NA	1,730	NS	**235**	**2,880**	**283**	**1,300**	NA	<200	**566[g]**	**638**
Antimony	6	<6.0	<5.0	<6.0	NS	NA	<6.0	NS	<6.0	<5.0	<6.0	<6.0	NA	<6.0	<6.0[g]	<6.0
Arsenic	3	**7.2**	**17**	<3.0	NS	NA	<3.0	NS	**18.3**	**10.4**	<3.0	**8.2**	NA	**3**	<3.0[g]	<3.0
Barium	6000	<200	597	<200	NS	NA	<200	NS	<200	<200	<200	<200	NA	<200	58.8[g]	<200
Beryllium	1	<1.0	NA	<1.0	NS	NA	<1.0	NS	<1.0	NA	<1.0	<1.0	NA	<1.0	<1.0[g]	<1.0
Cadmium	4	<3.0	NA	<3.0	NS	NA	6.4	NS	<3.0	NA	<3.0	<3.0	NA	<3.0	<4.0[g]	<3.0
Calcium		41,300	52,700	85,900	NS	NA	183,000	NS	46,000	146,000	23,800	34,800	NA	75,300	50,800[g]	18,200
Chromium	70	<10	10.3	<10	NS	NA	<10	NS	<10	<10	<10	<10	NA	<10	<10[g]	<10
Cobalt	100	<50	NA	<50	NS	NA	<50	NS	<50	NA	<50	<50	NA	<50	<50[g]	<50
Copper	1300	<10	<25	10.4	NS	NA	12	NS	<10	<25	<10	53.9	NA	<10	<25[g]	26.8
Iron	300	239	**16,200**	**2,370**	NS	NA	3,320	NS	**4,050**	**39,700**	**4,600**	**7,540**	NA	**8,680**	**7,250[g]**	**1,280**
Lead	5	<3.0	**10.2**	**8.3**	NS	NA	<3.0	NS	<9.0[c]	4.9	4.1	**6**	NA	<3.0	<5.0[g]	5.6
Magnesium		56,400	98,100	6,600	NS	NA	19,500	NS	<5,000	49,800	<5,000	<5,000	NA	28,100	8,160[g]	<5,000
Manganese	50	<15	***212***	***362***	NS	NA	499	NS	**57.7**	**879**	38.8	35.8	NA	**202**	**190[g]**	18.4
Mercury	2	<0.20	0.2	<0.20	NS	NA	<0.20	NS	<0.20	<0.20	<0.20	<0.20	NA	<0.20	<0.20[g]	<0.20
Nickel	100	<10	NA	**115**	NS	NA	24.5	NS	<10	NA	<10	<10	NA	<10	<40[g]	<10
Potassium		39,900	25,400	68,000	NS	NA	82,500	NS	63,400	188,000	<10,000	<10,000	NA	15,700	8,260[g]	<10,000
Selenium	40	<10	<5.0	<10	NS	NA	<10	NS	<10	<5.0	<10	<10	NA	<10	<10[g]	<10
Silver	40	<10	NA	<10	NS	NA	<10	NS	<10	NA	<10	<10	NA	<10	<5.0[g]	<10
Sodium	50000	**503,000**	**409,000**	**296,000**	NS	NA	91,700	NS	292,000	822,000	<10,000	12,700	NA	**85,300**	**78,000[g]**	**14,600**
Thallium	2	<2.0	NA	<2.0	NS	NA	<2.0	NS	<6.0[c]	NA	<2.0	<2.0	NA	<2.0	<2.0[g]	<2.0
Vanadium	60	<50	<50	<50	NS	NA	<50	NS	<50	<50	<50	<50	NA	<50	<10[g]	<50
Zinc	2000	<20	26.2	528	NS	NA	139	NS	27.6	28.2	<20	26.6	NA	<20	23.7[g]	39.3
Volatile Organic Compounds																
Acetone	6000	ND	ND	ND	NS	ND	ND	NS	ND	ND	8.8J	ND	ND	8.7J	ND	ND
Benzene	1	ND	0.71	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Bromodichloromethane	1	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
2-Butanone (MEK)	300	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Carbon disulfide	700	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Carbon Tetrachloride	1	ND	NR	NR	NS	NR	NR	NS	ND	NR	NR	NR	NR	NR	ND	ND
Chlorobenzene	50	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Chloroethane	5	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	70	0.57 J	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Dibromochloromethane	1	ND	NR	NR	NR	NR	NR	NS	ND	NR	NR	NR	NR	NR	ND	ND
1,2-Dibromoethane	0.03	ND	NR	NR	NR	NR	NR	NS	ND	NR	NR	NR	NR	NR	ND	ND
1,2 - Dichlorobenzene	600	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,3 - Dichlororbenzene	600	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,4 - Dichlorobenzene	75	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Dichlorodifluroromethane	1000	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethane	50	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichloroethane	2	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,1 - Dichloroethene	1	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
cis - 1,2 - Dichloroethene	70	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
trans - 1,2 - Dichlororethene	100	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,2 - Dichlororpropane	1	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	700	ND	1.6	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Freon 113		ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
2-Hexanone	300	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Isopropylbenzene	700	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Methylcyclohexane		ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Methyl Tert Butyl Ether (MTBE)	70	0.34 J	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
4-Methyl-2-pentanone (MIBK)		ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Methylene Chloride	3	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	1	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	600	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3 - Trichlorobenzene		ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,2,4 - Trichlorobenzene	9	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,1,1 - Trichloroethane	30	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2 - Trichloroethane	3	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	1	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl Chloride	1	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Xylenes (Total)	1000	ND	6	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Tert Butyl Alcohol	100	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Total Tic, Volatile	100/500	0.0	92.8	0.0	NS	0.0	0.0	NS	0.0	100	0.0	0.0	0.0	0.0	0.0	0.0
Semi-Volatile Organic Compounds																
Acenaphthene	400	ND	49	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Acenaphthylene	100	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Acetophenone	700	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Anthracene	2000	ND	0.64	ND	NS	ND	ND	NS	ND	ND	ND	0.193	ND	ND	ND	ND
Benzaldehyde		ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(a)anthracene	0.1	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	**0.304**	ND	ND	ND	ND
Benzo(a)pyrene	0.1	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(b)fluoranthene	0.2	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(g,h,i)perylene	100	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Benzo(K)fluoranthene	0.5	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Butyl Benzyl phthalate	100	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Biphenyl	400	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
4-Chloroaniline	30	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Carbazole		ND	10.7	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Caprolactam	5000	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Chrysene	5	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	0.113	ND	ND	ND	ND
3,3-Dichlorobenzidine	30	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
1,4-Dioxane	10	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Dibenzofuran		ND	7.6	ND	NS	ND	ND	NS	ND	22.8	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	700	6.5 B	ND	ND	NS	ND	ND	NS	7.5 B	ND	ND	ND	ND	ND	ND	6.9 B
Di-n-octyl phthalate	100	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Diethyl phthalate	6000	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Dimethyl phthalate	100	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
bis(2-ethylhext)phthalate	3	ND	ND	8.9	NS	ND	ND	NS	ND	ND	**94.3**	ND	**6.0**	ND	ND	ND
Dibenzo(a,h)anthracene	0.3	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Fluoranthene	300	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	0.269	ND	ND	ND	ND
Fluorene	300	ND	16.9	ND	NS	ND	ND	NS	ND	ND	ND	0.172	ND	ND	ND	ND
Hexachlorobenzene	0.02	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	0.2	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Isophorone	40	ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
2-Methylnaphthalene	30	ND	**67.5**	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Naphthalene	300	ND	141	ND	NS	ND	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND
Phenanthrene	100	ND	8	ND	NS	ND	ND	NS	ND	1.2	ND	0.726	ND	ND	ND	ND
Pyrene	200	ND	ND	ND	NS	ND	ND	NS	ND	ND	ND	0.302	ND	ND	ND	ND
1,2,4,5-Tetrachlorobenzene		ND	NA	ND	NS	ND	ND	NS	ND	NA	ND	ND	ND	ND	ND	ND
Total Tic, Semi-Volatile	100/500	12.0 (1) J	278.1	0.0	NS	0.0	35 (1) J	NS	0.0	65.8	39 (2) J	0.0	0.0	111 (2) J	4.5 (1) J	0.0

Table 2
Hess Corporation Former Port Reading Complex (HC-PR)
AOC 12: Smith Creek and Detention Basin
750 Cliff Road
Port Reading, Middlesex County, New Jersey

Summary of 2015 Groundwater Analytical Results

Client Sample ID:		NJ Groundwater Criteria (NJAC7:9C 7/07)1	NJ Interim Groundwater Criteria (NJAC 7:9C 11/15)2	AB-4D	PER-2	PER-2D	PER-3	PER-3D	PER-9	PER-9D	PER-9DD	PER-10	PER-10D
Lab Sample ID:				JC9218-4	JC9071-8	JC9071-9	JC9218-5	JC9218-6	JC9218-1	JC9218-2	JC9218-3	JC9218-7	JC9218-8
Date Sampled:				11/23/2015	11/20/2015	11/20/2015	11/23/2015	11/23/2015	11/23/2015	11/23/2015	11/23/2015	11/23/2015	11/23/2015
Matrix:				Ground Water	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water	Ground Water
GC/MS Volatiles (SW846 8260C)													
Benzene	ug/l	1	-	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	ND (0.24)	**2**
Bromodichloromethane	ug/l	1	-	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	**1.5**	ND (0.23)	ND (0.23)
Chlorobenzene	ug/l	50	-	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)	ND (0.19)
Chloroethane	ug/l	-	5	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)	ND (0.34)
1,2-Dibromoethane	ug/l	0.03	-	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	ND (0.23)	**0.25 J**	ND (0.23)	ND (0.23)
1,4-Dichlorobenzene	ug/l	75	-	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)
1,1-Dichloroethane	ug/l	50	-	0.46 J	ND (0.17)	0.35 J	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	0.33 J
1,2-Dichloroethane	ug/l	2	-	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)	ND (0.18)
1,1-Dichloroethene	ug/l	1	-	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)	ND (0.51)
cis-1,2-Dichloroethene	ug/l	70	-	ND (0.27)	ND (0.27)	0.39 J	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)	ND (0.27)
1,2-Dichloropropane	ug/l	1	-	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)	ND (0.39)
Methyl Tert Butyl Ether	ug/l	70	-	2.9	ND (0.24)	**693**	ND (0.24)	**77**	ND (0.24)	7.5	0.31 J	ND (0.24)	25.7
Tert Butyl Alcohol	ug/l	100	-	23	ND (2.8)	**204**	ND (2.8)	45.8	ND (2.8)	23.1	ND (2.8)	ND (2.8)	35
Tetrachloroethene	ug/l	1	-	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)	ND (0.40)
1,2,4-Trichlorobenzene	ug/l	9	-	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)
1,1,1-Trichloroethane	ug/l	30	-	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)	ND (0.25)
1,1,2-Trichloroethane	ug/l	3	-	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)	ND (0.21)
Trichloroethene	ug/l	1	-	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)	ND (0.22)
Vinyl chloride	ug/l	1	-	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)	ND (0.15)
Xylene (total)	ug/l	1000	-	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)	ND (0.17)
Total TIC, Volatile	ug/l	-	-	52 J	5.6 J	6.1 J	0	0	0	0	0	0	0
Total Alkanes	ug/l	-	-	0	0	0	0	0	0	0	0	0	0
GC/MS Semi-volatiles (SW846 8270D)													
Benzo(a)anthracene	ug/l	0.1	-	-	-	-	-	-	-	-	-	-	-
Benzo(g,h,i)perylene	ug/l	-	100	ND (0.31)	ND (0.31)	ND (0.33)	ND (0.31)	ND (0.31)	ND (0.31)	ND (0.31)	ND (0.32)	ND (0.31)	ND (0.31)
1,4-Dioxane	ug/l	-	0.4	**3.4**	ND (0.72)	ND (0.75)	ND (0.72)	ND (0.72)	ND (0.72)	ND (0.72)	ND (0.74)	ND (0.72)	**4.3**
bis(2-Ethylhexyl)phthalate	ug/l	3	-	ND (0.55)	2	ND (0.58)	ND (0.55)	ND (0.55)	ND (0.55)	**3.5**	ND (0.57)	ND (0.55)	ND (0.55)
2-Methylnaphthalene	ug/l	-	30	ND (0.29)	ND (0.29)	ND (0.31)	ND (0.29)	ND (0.29)	ND (0.29)	ND (0.29)	ND (0.30)	ND (0.29)	ND (0.29)
Benzo(a)anthracene	ug/l	0.1	-	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)
Benzo(a)pyrene	ug/l	0.1	-	ND (0.030)	ND (0.030)	ND (0.031)	ND (0.030)	ND (0.030)	ND (0.030)	ND (0.030)	ND (0.030)	ND (0.030)	ND (0.030)
Benzo(b)fluoranthene	ug/l	0.2	-	ND (0.021)	ND (0.021)	ND (0.022)	ND (0.021)	ND (0.021)	ND (0.021)	ND (0.021)	ND (0.022)	ND (0.021)	ND (0.021)
Benzo(k)fluoranthene	ug/l	0.5	-	ND (0.019)	ND (0.019)	ND (0.020)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.019)	ND (0.020)	ND (0.019)	ND (0.019)
Dibenzo(a,h)anthracene	ug/l	0.3	-	ND (0.035)	ND (0.035)	ND (0.037)	ND (0.035)	ND (0.035)	ND (0.035)	ND (0.035)	ND (0.036)	ND (0.035)	ND (0.035)
Hexachlorobenzene	ug/l	0.02	-	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)	ND (0.015)
Indeno(1,2,3-cd)pyrene	ug/l	0.2	-	ND (0.031)	ND (0.031)	ND (0.033)	ND (0.031)	ND (0.031)	ND (0.031)	ND (0.031)	ND (0.032)	ND (0.031)	ND (0.031)
Total TIC, Semi-Volatile	ug/l	-	-	64.5 J	50.7 J	0	0	10 J	0	15.7 J	0	67.2 J	54.5 J
Total Alkanes	ug/l	-	-	0	0	0	0	0	0	0	0	0	0
Metals Analysis													
Aluminum	ug/l	200	-	**1290**	**632**	<200	<200	<200	**2610**	<200	<200	<200	<200
Antimony	ug/l	6	-	<6.0	<6.0	<6.0	<6.0	<6.0	<6.0	<6.0	<6.0	<6.0	<6.0
Arsenic	ug/l	3	-	**13.5**	<3.0	<3.0	<6.0 [h]	<3.0	**5.6**	<3.0	<3.0	**4.7**	<3.0
Arsenic	ug/l	3	-	-	-	-	-	-	-	-	-	-	-
Barium	ug/l	6000	-	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200
Beryllium	ug/l	1	-	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Cadmium	ug/l	4	-	<3.0	<3.0	<3.0	<3.0	<3.0	<3.0	<3.0	<3.0	<3.0	<3.0
Cadmium	ug/l	4	-	-	-	-	-	-	-	-	-	-	-
Calcium	ug/l	-	-	149000	23000	113000	104000	82400	42400	98600	48700	66200	103000
Chromium	ug/l	70	-	-	-	-	-	-	-	-	-	-	-
Chromium	ug/l	70	-	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Iron	ug/l	300	-	**2090**	**2460**	<100	**1010**	131	**5550**	185	**896**	**2040**	123
Lead	ug/l	5	-	<30 [i]	**5.5**	<3.0	<6.0 [h]	<3.0	3.3	<3.0	<3.0	<3.0	<3.0
Manganese	ug/l	50	-	21.4	**120**	29.4	**253**	**194**	**70.8**	48.5	**63.6**	**98.3**	**656**
Nickel	ug/l	100	-	<100 [i]	<10	<10	<10	<10	<10	<10	<10	<10	15.2
Sodium	ug/l	50000	-	**2230000**	29600	**69800**	**1540000**	**1110000**	**198000**	**227000**	**126000**	**160000**	**1320000**
General Chemistry													
Nitrogen, Ammonia	mg/l	3	-	**6.1**	0.56	<0.20	0.22	**8.1**	<0.20	0.88	<0.20	0.94	**18.7**

APPENDICES

QUALITY ASSURANCE PROJECT PLAN

AOC 12: Smith Creek and Detention Basin
Hess Corporation – Former Port Reading Complex (HC-PR)
750 Cliff Road
Port Reading, Middlesex County, New Jersey
NJDEP PI# 006148
ISRA Case No. E20130449
EPA ID No. NJD045445483

PREPARED FOR:

Hess Corporation
Trenton-Mercer Airport
601 Jack Stephan Way
West Trenton, New Jersey 08628

PREPARED BY:

Earth Systems

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Distribution List:

John Virgie, LSRP – Earth Systems, Inc.
LSRP of Record
1625 Route 71
Wall, New Jersey
732-739-6444
jvirgie@earthsys.net

Amy Blake, Senior Project Manager
Earth Systems, Inc.
1625 Route 71
Wall, New Jersey
732-739-6444
ablake@earthsys.net

Paul Simms – Alpha Analytical
8 Walkup Drive
Westborough, Massachusetts 01581
508-898-9220
psimms@alphalab.com

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INTRODUCTION

This Quality Assurance Project Plan (QAPP) was prepared by Earth Systems, Inc. (Earth Systems) for Hess Corporation, who is conducting remedial investigation (RI) activities at an environmental area of concern designated as AOC 12: Smith Creek and Detention Basin located at 750 Cliff Road, Port Reading (Woodbridge Township), Middlesex County, New Jersey (Property or site).

The purpose of this QAPP is to ensure that scientific data are acquired according to established methods and procedures designed to obtain results that are objective, true, repeatable, and of known accuracy. Specifically, this QAPP provides guidance and specifications to ensure that RI activities are planned and executed in a manner consistent with the Quality Assurance Objectives (QAO's) stated below:

- Field determinations and analytical results are valid through adherence to New Jersey Department of Environmental Protection (NJDEP) field procedures, NJDEP-approved analytical protocols, and calibration and preventive maintenance of equipment;
- Samples are identified and controlled through sample tracking systems and chain of custody procedures;
- Records are retained as documentary evidence of field activities and observations;
- Samples are collected and analytical data are validated in accordance with the NJDEP requirements; and
- Evaluations of the data are accurate, appropriate, and consistent throughout the project.

The contents of this QAPP are based on the NJDEP requirements as stated in the NJDEP Technical Requirements for Site Remediation and the Quality Assurance Project Plan Technical Guidance (Version 1.0, April 2014). This QAPP includes the following components:

- Problem Definition/Background;
- Project/Task Description;
- Project/Task Organization;
- Data Quality Objectives and Criteria for Measurement Data;
- Historical and Secondary Information/Data;
- Investigative Process Design;
- Field Instrumentation/Equipment Calibration and Frequency;
- Inspection/Acceptance of Supplies and Consumables;
- Sample Handling and Custody Requirements;
- Field Storage and Transport Procedures;
- Sample Containers, Preservation, and Holding Times;
- Analytical Methods Summary Table;
- Project Compounds and Analytical Summary;
- Analytical Quality Control;
- Laboratory Deliverables;
- Data and Records Management;
- Data Verification and Usability; and
- Corrective Action Processes.

As specific conditions and additional information warrant, this QAPP will be amended or revised to include site-specific quality assurance/quality control procedures.

Quality Assurance Project Plan
AOC 12: Smith Creek and Detention Basin
Hess Corporation – Former Port Reading Complex
750 Cliff Road
Port Reading, Middlesex County, New Jersey

1. Project Definition / Background

Project Definition

The property is owned by Hess Corporation and is located at 750 Cliff Road, Port Reading, New Jersey. This QAPP only addresses the investigation of AOC 12: Smith Creek and Detention Basin. The detention basin present on the Site was created between 1966 and 1969. The detention basin is approximately 800 feet by 600 feet in size with a depth of approximately 5 to 6 feet. Based on a review of historic aerial photographs, it appears that the northern portion of Smith Creek and its tributaries were filled between 1954 and 1957. By 1963, only the southern portion of Smith Creek was present on the Site. All northern portions of the Creek were filled-in, excluding the northern drainage ditch. In 1972, Smith Creek was no longer present on the Site and the detention basin was no longer directly connected to the off-site portion of Smith Creek. Smith Creek is approximately 125-175 feet wide and approximately 5,000 feet long from where the Creek begins, to where it eventually discharges into the Arthur Kill.

The Site detention basin and off-site portions of Smith Creek have been potentially impacted due to former releases, the presence of historic fill, and historic operations on the Site. Contamination migration pathways include overland flow, direct discharges, and groundwater migration. Therefore, sediment and surface water sampling are required to assess and delineate any impacts. Based on groundwater monitoring results, the contaminants of concern include benzene, MTBE, TBA, arsenic, lead, and ammonia. The arsenic and lead groundwater impacts are likely due to the presence of historic fill across the Site.

The overall project goals and objectives are summarized below:

- Sediment & Surface Water Investigation of detention basin and off-site Smith Creek;
- Additional groundwater monitoring.

The analytical data shall be used to determine if further soil and groundwater investigation is required. These decisions shall be made following receipt of all analytical data associated with the investigation. Data users for the project include the person responsible for conducting the remediation, the environmental consultant, and ultimately, the NJDEP.

2. Project / Task Description

Sediment and surface water samples will be collected from both the Site detention basin and off-site Smith Creek. All sediment samples and surface water samples will be collected in accordance with the FSPM and applicable guidance. Sample collection will begin downstream and proceed to the upstream sample locations in order to minimize disturbance and to ensure that the samples are representative. All sediment

and surface water samples will be collected at the same location; the surface water sample will be collected prior to collection of the sediment sample. The type of equipment utilized for collecting the sediment sampling will depend on the location of the sample. Sampling equipment will be selected accordingly in order to achieve the data quality objectives and to ensure the health and safety of sampling personnel.

Prior to sampling; the size, depth, and flow of the water body will be recorded in a dedicated field book. In addition, the current tidal stage of the adjacent Arthur Kill will be recorded. A calibrated water quality meter will be utilized to record pH, temperature, conductivity, salinity, dissolved oxygen, ORP, and turbidity for all surface water samples.

Depending on access and the location of the proposed sampling locations, surface water samples will either be collected as a direct grab sample or with a depth sampler, such as a Kemmerer depth sampler. For grab samples, a sample container will be placed just below the surface of the water and allowed to fill to the required sampling volume. If necessary, surface water sampling equipment may be used for grab sampling, such as a pond sampler. If a water sample is required at depth, the appropriate sampling device will be used to isolate the desired interval and the depth of the sample will be recorded in the field book. If sampling equipment is used, it will be decontaminated in between sampling locations. Surface water samples collected using sampling equipment will be transferred directly into the laboratory provided glassware.

Sediment is a heterogeneous material and the correct sampling equipment and collection methods must be utilized to ensure that a representative sample is collected. Care must be taken to minimize disturbance and the loss of fines as the sample is retrieved through the water column. Any water that is collected along with the sediment sample will be transferred to the laboratory provided jar and will not be decanted off the sample. Sediment samples will be biased towards observed depositional areas and collected using a sediment coring device or dredge. A sample will be collected in the 0 to 6-inch surficial interval as well as from the 6 to 12-inch interval below grade. Observations regarding sediment color, particle size, and any evidence of impacts will be recorded in a dedicated field book.

In addition to the above sampling, the frequency of groundwater monitoring will be increased to quarterly for one year in order to evaluate possible seasonal fluctuations on groundwater impacts.

Sample results shall be compared to the applicable remediation standards and a conclusion shall be made, based on the comparison, as to whether the Area of Concern (AOC) requires further investigation / action or no further investigation / action is required.

The applicable regulatory quality standards to this phase of investigation are:

- NJDEP Residential and Non-Residential Soil Remediation Standards (SRS)
- NJDEP Groundwater Quality Standards (GWQS)
- NJDEP Ecological Screening Criteria
- NJDEP Surface Water Quality Criteria

3. Project / Task Organization

The NJDEP's "Quality Assurance Project Plan Technical Guidance" recommends that the QAPP include an organizational chart identifying key personnel and/or organizations showing relationships and lines of communication. As stated in Section 5 of the guidance, not all elements of the QAPP may need the same level of detail, which should be based on a graded approach depending on the complexity of the project and

the intended use of the data. In this regard, since the number of personnel and organizations is relatively small, the relationships can be described rather than depicted in a chart.

Project Team

The Licensed Site Remediation Professional (LSRP) is John Virgie of Earth Systems. He also serves as the central point of communication with all other individuals and organizations associated with this project. He is responsible for implementing the Quality Assurance Project Plan and coordinating the site investigation activities. He can be reached at (732) 739-6444.

The Project Manager is Ms. Amy Blake of Earth Systems. She is responsible for coordinating the site investigation activities in the field and tabulating/interpreting the analytical data once received. She can be reached at (732) 739-6444.

The Health and Safety Officer for Earth Systems is Mr. Michael Piegaro. He can be reached at (732) 739-6444.

Laboratory: Alpha Analytical: 8 Walkup Drive, Westborough, Massachusetts 01581 (Contact: Mr. Paul Simms @ psimms@alphalab.com).

Special Training Needs/Certification

Training needs and certifications of field oversight include requirements to have completed the OSHA 40-Hour training with annual 8-hour refresher training in accordance with 29 CFR 1910.120 (Hazardous waste operations and emergency response). In addition, site workers must have a TWIC card and at least one person on-site must have completed Buckeye Person-In-Charge (PIC) training.

The site investigation activities are being conducted under the oversight of an LSRP.

Special training is required to operate laboratory equipment and conduct laboratory analyses. Laboratory certification is established at N.J.A.C. 7:18.

4. Data Quality Objectives and Criteria for Measurement Data

Data quality objectives ("DQOs") are qualitative and quantitative statements that are developed in the first six (6) steps of the DQO process. DQOs define the purpose of the data collection effort, clarify what the data should represent to satisfy this purpose, and specify the performance requirements for the quality of information to be obtained from the data.

In accordance with Section 5.4 of the NJDEP's "Quality Assurance Project Plan" technical guidance, the development of the data quality criteria can be developed through the formal DQO process described in the EPA document titled "Guidance for the Data Quality Objectives Process", EPA/600/R-96/055. For most projects, however, a less iterative process is normally used to develop the project-specific DQOs.

Data of Known Quality Protocols ("DKQP") describe specific laboratory quality assurance and quality control procedures which, if followed, will provide data of known and documented quality (i.e. scientific reproducible and reliable data). When data of known quality ("DKQ") is obtained, an evaluation of the

data with respect to its intended purpose can be made. To this end, a NJDEP-certified laboratory must be used to analyze samples whenever possible.

Typical DQOs are often expressed in terms of data quality indicators ("DQIs") including precision, accuracy, representativeness, comparability, completeness and sensitivity (also known as the "PARCCS" parameters). These measures of performance are discussed in detail below.

Precision

Precision is the measure of agreement among repeated measurements of the same property under identical or substantially similar testing conditions. The investigator will determine the precision of the data by:

- Using the same analytical methods to perform repeated analyses on the same sample (laboratory or matrix duplicates);

Precision for laboratory and field measurements can be expressed as the relative percent difference ("RPD") between two duplicate determinations or percent relative standard deviation ("%RSD") between multiple determinations.

Accuracy

Accuracy is the degree of agreement of a measured value and an accepted reference or true value. The difference between the measured value and the reference or true value includes components of both systematic error (bias) and random error (precision). It should be noted that precise data may not be accurate data. Accuracy can be expressed as a percent recovery or percent deviation of the measurement with respect to its known or true value.

The accuracy will be determined through establishing acceptance criteria for spike recoveries (e.g., surrogate recoveries, laboratory control sample recoveries, matrix spike recoveries, reference material recoveries etc.) or allowable deviations for calibration (e.g., %RPD for calibration verification). Acceptance criteria for matrix spike measurements are expressed as a percent recovery and are usually specified in the analytical method (or laboratory SOP, as applicable). Various blank samples (laboratory or field) may also be used to assess contamination of samples that may bias results high. Accuracy in the field shall be assessed through the adherence to sample collection, handling, preservation, and holding time requirements.

Representativeness

Representativeness is a qualitative measurement that describes the extent to which analytical data represent the site conditions. In almost every project, the investigator will not be able to measure the whole system, process, or situation of interest. Instead, the investigator will choose sample locations, quantities, and analyses in order to capture a sufficiently broad and/or weighted view of the situation.

Representativeness in the laboratory is ensured by using the proper analytical procedures, appropriate methods, and meeting sample holding times. Following the detailed requirements outlined in the EPA methods and the laboratory SOPs will maximize the representativeness of the laboratory data.

Comparability

Comparability is a qualitative term that expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Comparability is defined as the extent to which data from one data set can be compared directly to similar or related data sets and/or decision-making standards.

Historical data should be evaluated to determine whether they may be combined with data being collected in present time. Comparability should discuss comparisons of sample collection and handling methods, sample preparation, and analytical procedures, holding times, stability issues and QA protocol.

Comparability in the laboratory is dependent on the use of recognized methods and approved laboratory SOPs. Comparability in the field is dependent upon adherence to the sampling methodology and that the proper preservation techniques are used.

Completeness

Completeness is a measure of the amount of usable data collected compared to the amount of data expected to be obtained. Three measures of completeness are defined as:

- Sampling completeness, defined as the number of valid samples collected relative to the number of samples planned for collection;
- Analytical completeness, defined as the number of valid sample measurements relative to the number of valid samples collected; and
- Overall completeness, defined as the number of valid sample measurements relative to the number of samples planned for collection.

Sensitivity

Sensitivity refers to the ability of an analytical procedure to quantify an analyte at a given concentration. The sensitivity requirements should be established such that the laboratory method Reporting Limits ("RLs") are at or below the relevant and applicable regulatory limits for each Contaminant of Concern ("COC") for the project. For the purpose of SRP projects:

- The RL for a specific substance when determining the extent and degree of polluted soil, groundwater, or sediment from a release. For the purpose of this document, the RL is defined as:
  - Organics, the lowest initial calibration standard as adjusted for the dilution factor, sample weight/volume, and moisture content;
  - Inorganics, the concentration of that analyte in the lowest level check standard (which could be the lowest calibration standard in a multi-point calibration curve).

Methods for analysis have been chosen to meet the sensitivity requirements for a project (e.g., compound-specific and matrix-specific). If however, the laboratory RLs exceed the project sensitivity requirements (i.e., the RL is above the relevant and applicable regulatory standard), the analytical methods may need to be adjusted (e.g., analysis conducted using a more sensitive method or sample preparation and analysis features adjusted to gain sensitivity) and/or the project objectives may need to be adjusted (i.e., certain COCs may not be able to be screened out during this phase of the evaluation).

5. Historical and Secondary Information / Data

The potential sources of data for any project include both historical data (i.e. data not collected by the current investigator) and secondary data (i.e. data that were collected for a different purpose than that for which they are now being used). Historical data should be evaluated for applicability to current project objectives. Secondary data should be assessed to determine if the quality of the data is sufficient for the current project objectives and meets comparability criteria (it is not sufficient that the secondary data were produced by a reliable source or a known environmental monitoring project with an approved QAPP).

6. Investigation Process Design

A description and justification of the investigation design should include, for each area of interest:

- The COCs or other parameters of interest
- The number of anticipated investigation points and how and why they will be selected including a site map depicting proposed sample locations
- Method of obtaining/determining locational information (such as the use of GPS instrumentation)
- Factors which could affect the variability of the data such as physical obstructions, seasonal variations, tidal influences, soil profile changes, weather-related variation, and process variation within the source
- Design basis i.e. probability based or judgment based
- Results comparison (i.e. versus previous data, regulatory standards, reference population, etc.)
- Matrices to be monitored including any special sampling requirements
- Monitoring frequency (if applicable)
- Heterogeneity or homogeneity of the matrix
- Appropriateness of composite samples
- Required quality control samples

The investigative process design is based generally on the following:

- The Technical Requirements for Site Remediation N.J.A.C. 7:26E.
- The NJDEP's "Field Sampling Procedures Manual (FSPM)" dated August 2005.

7. Field Quality Control

Field equipment cleaning / decontamination are not expected to be required as all field equipment shall be dedicated to each individual sample. However, if decontamination is required, equipment will be cleaned using a detergent wash followed by a tap water rinse. The final step will be to rinse the equipment using distilled/deionized water.

8. Field Instrumentation / Equipment Calibration and Frequency

Field instrumentation/equipment that will require calibration includes a photoionization detector (PID) and water quality meter. Calibration and routine maintenance procedures are presented in the User's Manual. Documentation of the maintenance and calibration records is stored at the office or in the field logbook.

9. Inspection / Acceptance of Supplies and Consumables

Critical supplies or consumables (e.g., pre-cleaned containers, pre-preserved containers, tubing, etc.) shall be inspected for visible indications of contamination and damage and, if none are identified, then the supplies/consumables shall be accepted for use.

10. Sample Handling and Custody Requirements

Sample handling shall be as specified in Section 2.5.5.1 of the FSPM and Section 4.6.2.2 of the NJDEP's "Data Quality Assessment and Data Usability Evaluation Technical Guidance", Version 1.0, dated April 2014. Specifically, samples shall be maintained on-site for no more than two (2) consecutive days, and shall be delivered to the laboratory within one (1) day of shipment from the field.

The chain of custody procedure to be utilized in the field is specified in Section 2.3.6 of the FSPM. The chain of custody procedure to be used in the laboratory shall be in accordance with Section 2.3.6 of the FSPM as well as the laboratory's standard operating procedure.

11. Field Storage and Transport Procedures

Samples shall remain in direct site and in the custody of field personnel at all times until transfer to the laboratory.

12. Sample Containers, Preservation, and Holding Times

Sample containers, preservation, and holding times are specified on Table 1.

13. Analytical Methods Summary Table

Analytical methods are summarized on Table 1.

14. Project Compounds and Analytical Summary

All surface water samples will be analyzed for TCL VO+15, TCL SVOCs +15, TAL Metals, dissolved metals, hardness, alkalinity, and chloride. All sediment samples will be analyzed for TCL VO+15, TCL SVOCs+15, TAL metals, Total Organic Carbon (TOC) pH, Extractable Petroleum Hydrocarbons (EPH) and grain size. Groundwater samples will be collected and analyzed for TCL VOC+15, TAL Metals, and TCL Base Neutrals plus TICs. The project action limits are the NJDEP's SRS, GWQS, Ecological Screening Criteria, and Surface Water Quality Criteria. The analytical methods chosen can meet the DQOs of the project.

Analytical sensitivity requirements include the use of instruments or methods to detect the contaminants of concern at or below the action limits. The RLs are expected to be below the applicable regulatory standards. NJDEP and EPA methods were selected to achieve the action limits. Laboratories may need to adjust RLs based on dilutions, sample sizes, extract/digestate volumes, percent solids and cleanup procedures. Sensitivity will be maximized by following the NJDEP and EPA methods or laboratory SOPs utilizing experienced, trained laboratory personnel and by conducting laboratory audits.

15. Analytical Quality Control

Quality assurance and quality control ("QA/QC") requirements for analysis are specified in the most recent version of the document titled "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", prepared by EPA. The laboratory may also have QA/QC procedures in addition to those specified by the test method (Appendix 1).

16. Laboratory Deliverables

The laboratory deliverable format to be used for this project shall be the reduced laboratory deliverable format as described in Appendix A of N.J.A.C. 7:26E. The laboratory shall also generate Hazsite files and spreadsheets of the analytical results.

17. Data and Records Management

The recording media for the project will be both paper and electronic. The project will implement proper document control procedures for both, consistent with NJDEP's Quality Management Plan. For instance, hand-recorded data records will be taken with indelible ink, and changes to such data records will be made by drawing a single line through the error with an initial by the responsible person. The Project Manager will have ultimate responsibility for any and all changes to records and documents. Similar controls will be put in place for electronic records.

The Quality Assurance Coordinator shall retain all updated versions of the QAPP and be responsible for distribution of the current version of the QAPP. The Quality Assurance Coordinator and the Project Manager will approve periodic updates. The Project Manager shall retain copies of all management reports, memoranda, and all correspondence between the parties identified in Section 3.

Project data shall be stored in the Project Manager's office. Laboratory records management is described in Appendix 1.

18. Data Verification and Usability

The procedure for review (verification and usability procedures) including data assessment versus stated data quality objectives of the investigation is specified in the NJDEP's "Data Quality Assessment and Data Usability Evaluation Technical Guidance", Version 1.0, dated April 2014.

19. Corrective Action Processes

Corrective action in the field may be needed when the work plan is modified (i.e. number or locations of samples) or when sampling procedures and/or field analytical procedures require modification due to unexpected conditions. The corrective action may be implemented at the time the determination is made in the field or may be implemented later, depending on the circumstances. Any corrective actions taken shall be documented in the field logbook and in the technical report.

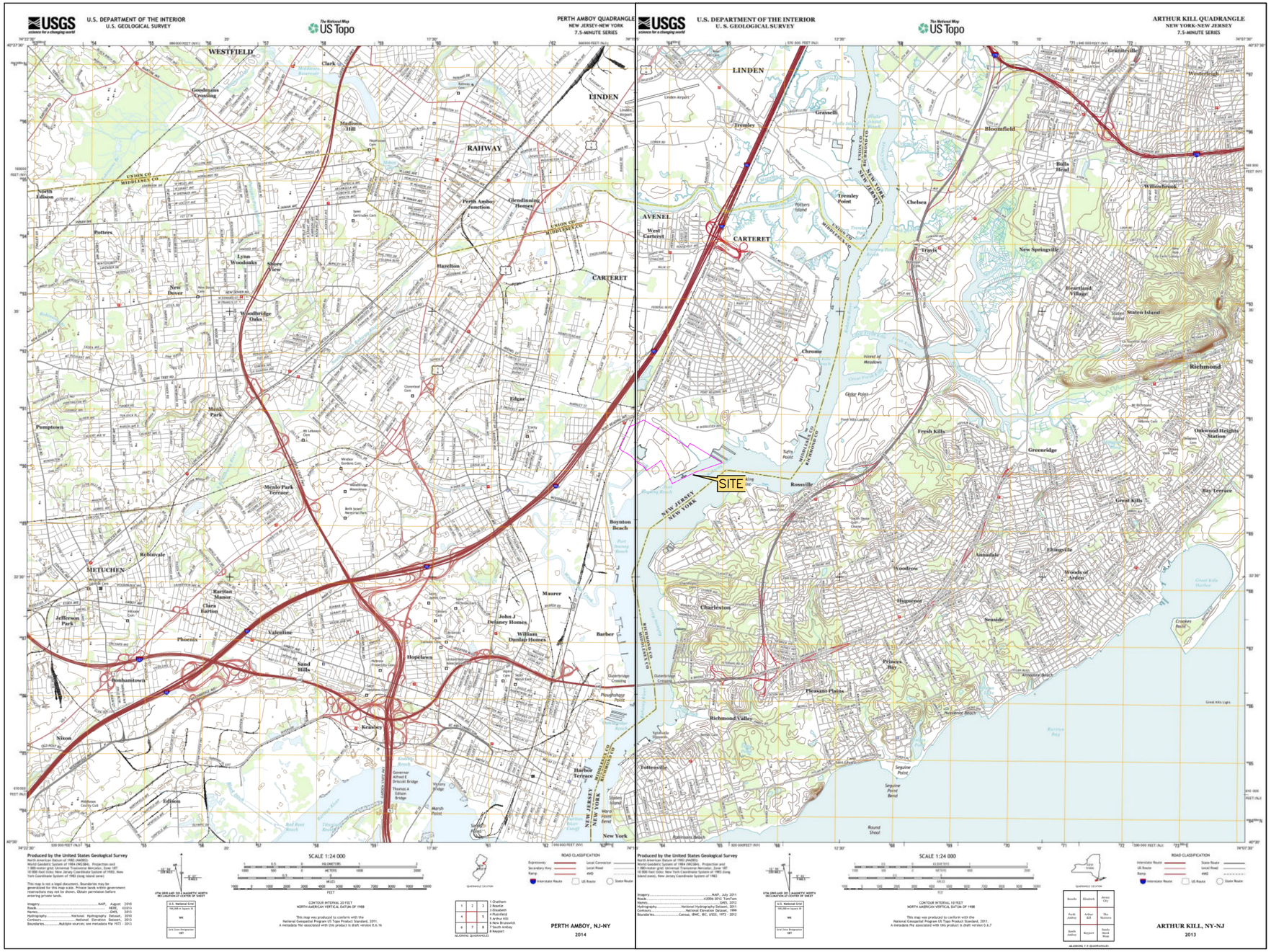
Corrective actions in the laboratory may be needed when Non-Conformances occur. The laboratory shall implement and document corrective actions in accordance with the laboratory SOP.

Table 1: Analytical Methods / Quality Assurance Summary Table

TABLE 1
Analytical Methods/Quality Assurance Summary Table
AOC 12: Smith Creek and Detention Basin, Hess Corporation - Former Port Reading Complex, Port Reading, Middlesex County, New Jersey

Matrix type	Number of Samples	Number of Blanks	Number of Duplicates	Analytical Parameters	Analytical Methods	Sample Preservation	Sample Container & Volume	Permissible Holding Time
Sediment	33	1 per day of sampling	0	Extractable Petroleum Hydrocarbons	NJ EPH	<6ºC HC to pH<2	1x4 ounce clear	14 days
Sediment	33	1 per day of sampling	0	Volatile Organic Compounds	8260C	4ºC methanol	EnCore	14 days
Sediment	33	1 per day of sampling	0	Base Neutrals	FW846 SW846, 8270D	4ºC N/A	8 oz.	14 days
Sediment	33	1 per day of sampling	0	Metals	SW846 7470A/ SW846 6010C	4ºC N/A	8 oz.	28 for Mercury/ 6 months
Sediment	33	1 per day of sampling	0	Total Organic Carbon (Lloyd Kahn)	Lloyd Kahn 1998	4ºC N/A	8 oz.	14 days
Sediment	33	1 per day of sampling	0	Grain Size	422-63	N/A	950 ml Amber Glass	N/A
Groundwater	48	4	0	Volatile Organic Compounds	8260	4ºC, HCl	Clear glass 40 ml	14 days
Groundwater	48	4	0	Metals	SW846/6010	HNO3 to pH<2	500-ml Amber	6 months
Groundwater	48	4	0	Base Neutrals	8270	<6ºC	2 x 1000-ml Amber	7 days extraction/40 days holding time
Surface Water	35	1 per day of sampling	0	Volatile Organic Compounds	8260	4ºC, HCl	Clear glass 40 ml	14 days
Surface Water	35	1 per day of sampling	0	Metals	SW846/6010	HNO3 to pH<2	500-ml Amber	6 months
Surface Water	35	1 per day of sampling	0	Base Neutrals	8270	<6ºC	2 x 1000-ml Amber	7 days extraction/40 days holding time
Surface Water	35	1 per day of sampling	0	Hardness, alkalinity, chloride	SM2340 C-11 2320-B11 20 4500 CL-C	HNO3 N/A N/A	Plastic 500 ml Plastic 250 ml Plastic 250 ml	180 days 14 days 25 days

Figure 1: Site Location Map



USGS MAP

Hess Corporation Former Port Reading Complex (HC–PR)
750 Cliff Road
Port Reading, New Jersey

Earth Systems

Figure 1

09/15 FIG1

Figure 2: Location of Area of Concern

NORTH

LEGEND

SURFACE WATER SAMPLE

SURFACE WATER AND SEDIMENT SAMPLE

FILE: F:\CLIENTS\E\EARTHSYSTEMS\HESS\PINNACLE\SED_SAMPLES.DWG, DATE: 02/05/2016 12:58:33PM

Earth Systems

SCALE:

0 600

SCALE: 1"=600'

DATE: 2/5/16

DRAWN BY: AG

CHECKED BY: JC

FILENAME: SED_SAMPLES

LAYOUT: SITE PLAN

PROJECT:

HESS CORPORATION

FORMER PORT READING COMPLEX (HC-PR)
750 CLIFF ROAD
PORT READING, NEW JERSEY

TITLE:

AOC-12 - SEDIMENT AND SURFACE WATER SAMPLE MAP

FIGURE NO.

2

Appendix 1: Laboratory Quality Systems Manual

Quality Systems Manual

Alpha Analytical, Inc.

D/B/A

Alpha Analytical
Eight Walkup Drive
Westborough, MA 01581-1019
Telephone: (508) 898-9220
Facsimile: (508) 898-9193

Alpha Analytical
Woods Hole Labs Facility
320 Forbes Blvd
Mansfield, MA 02048
Telephone: (508) 822-9300
Facsimile: (508) 822-3288

Email: info@alphalab.com
Web site: www.alphalab.com

President: Mark Woelfel, 508-439-5115
Quality Assurance Officer: James Todaro, Ext 508-439-5101
Laboratory Technical Manager (Director) Westboro: Mitch Ostrowski 508-439-5175
Laboratory Technical Manager (Director) Mansfield: Joseph Watkins, 508-844-4125
Laboratory Technical Manager (Director) Air-Mansfield: Andy Rezendes, 508-844-4181

1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our customers with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and electronic data deliverables must be available to the customer.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional contamination to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honesty and integrity with which each chemist, courier, login staff member, or office staff member performs their tasks. The customer or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical will vigorously pursue its mission into the next millennium.

Mark Woelfel

President

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3 Introduction

The Quality Systems Manual, referred to as Corporate Quality Systems Manual (CQSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, customers and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet customer needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as an uncontrolled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly to the QSM and SOPs.

All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) 2009 TNI standards, applicable EPA requirements andstandards.

Twenty-five (25) sections comprise the QSM. Related quality documentation including the listing of SOPs, forms, floor plan, equipment, personnel and laboratory qualifications are available. The QSM sections provide overview descriptions of objectives, policies, services and operations.

3.1 Scope

The QSM describes the requirements of the Laboratory to demonstrate competency in the operations for performing environmental tests for inorganic, organic, air and microbiological testing. The basis for the environmental tests is the methods found in documents published by the United States Environmental Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, and other procedures and techniques supplied by customers.

The QSM includes requirements and information for assessing competence and determining compliance by the laboratory to the quality system. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan the laboratory demonstrates achievement of the customer specified requirements through its documented processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and customers use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) TNI 2009 standards, applicable EPA requirements, regulations, guidance, and technical standards. This Quality Systems Manual, laboratory Standard Operating Procedures (SOPs),

and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by NELAC and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operations Director, Laboratory Technical Manager and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our customer's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accordance with NELAC and other regulatory programs, from a National Institute of Standards and Technology (NIST)-approved PT provider for the analytes established by EPA for water samples, and for other analytes and matrices. The specific analytes and matrices analyzed are based on the current scope of the laboratory services as documented in the laboratory SOPs and state certifications.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstrated according to the procedure described in SOP/ 1739 prior to use.

Alpha Analytical has developed a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program include: internal proficiency testing, electronic data audits and post-analysis data review by the QA Officer; a program to improve employee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequences. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form upon commencement of employment and each year following. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation. All staff are required to report any suspected unethical conduct to management. Management will then investigate and determine if the situation was considered unethical and will take appropriate action as described in the Alpha Ethics policy.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory Technical Manager to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory Technical Manager to take appropriate action to prevent recurrence.

3.3 References

External reference documents are available electronically in the Qualtrax system for staff to access the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the electronic files in the Qualtrax system. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

3.4 Definitions

Appendix A lists the definitions as adopted by the laboratory. The definitions are from the 2009 TNI standards.

4 Organization and Management

4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

Consulting firms,
Engineering firms,
Waste Management Companies,
Industrial sites,
Municipal agencies
Department of Defense projects.

4.2 Organization

The laboratory operates a quality system approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting customer specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise the integrity of the reporting of the final data. See Appendix B for Organizational Chart.

The President is responsible for directing all areas of the company. The following job functions report to the President:

Operations Manager
Quality Assurance Officer
Customer Services Manager
Marketing / Business Development / Sales
Financial Services
Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

- Laboratory Technical Manager(s)
- Department Managers

The Laboratory Technical Manager(s) is(are) responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

The organics managers direct personnel in the organics extraction and instrumental laboratories.

The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.

The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff and reports directly to the President and has defined responsibility and authority for ensuring that the quality system is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Quality Systems Manual and reporting to the Laboratory Technical Manager and Senior Management the status of the quality program. The QAO oversees the Quality Systems Specialists and is responsible for oversight and/or review of quality control data and function independently from laboratory operations.

The Customer Services Manager is responsible for customer interactions, project coordination and laboratory personnel notification of project requirements.

The Marketing, Business Development and Sales personnel are responsible for increasing the volume of work from current customers and adding new customers to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Technical Manager, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The Controller is responsible for maintaining and reporting on the financial status of the company. The Controller directs financial personnel on proper accounting procedures and maintaining the list of approved suppliers and subcontractors. The Controller reports directly to the President.

The Human Resource Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak workloads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Technical Manager is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of NELAC the Lead Laboratory Technical Manager is the Laboratory Technical Manager. The deputies for the Lead Technical Manager are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory Technical Manager meets the requirements specified in the Section 4.1.7.2 Volume 1, Module 2 of the 2009 TNI standards. If the Laboratory Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff member meeting the qualifications of Laboratory Technical Manager will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Manager's absence exceeds 35 consecutive calendar days.

4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of sample receipt. Standard turnaround for all other customers is ten business days from time of sample receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any special arrangements for rush or expedited turnaround time. The basis for data quality depends on customer, regulation and method performance criteria. Accuracy, precision, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for protecting customer confidentiality and proprietary rights. The review occurs during orientation and ethics training. It is the policy of the laboratory to release data to the customer authorized contact. Personnel assigned the duties of interacting with customers review project files and discuss data related only to the project. Personnel whose duties do not include routine customer contact must check with the customer service manager before discussing data with regulators or third parties

5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in order to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its customers with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and caliber in the field of analytical chemistry. This and state-of-the-art instrumentation and automation combine to insure data of known and documented quality.

5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distribution of the Quality Systems Manual. Management reviews and authorizes the manual. Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization of the Quality Systems Manual is documented electronically in Qualtrax. Updates of this manual occur at any time throughout the year. Document control procedures (SOP1729) apply to the distribution of the Quality Systems Manual. Controlled copies of the manual are maintained electronically within Qualtrax. Persons or organizations outside of Alpha Analytical may receive uncontrolled copies. Copies are distinctly indicated “Uncontrolled Documents” within the footer of each page.

5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are conducted by qualified QA Specialists and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

- Verification that adequate written instructions are available for use;
- Analytical practices performed in the laboratory are consistent with SOPs;
- The quality control practices are applied during production;
- Corrective actions are applied as necessary;

Deviations from approved protocols are occurring only with proper authorization and documentation;

Reported data is correct and acceptable for reporting;

SOPs, quality records, analytical records, electronic data files are maintained properly; and

Personnel training files and records are satisfactory and current.

Before a scheduled internal audit, the assigned auditor reviews checklists, if used, and/or the SOP specific to the area. The checklist may be from an external source or prepared by the auditor. . After the audit, the auditor submits a summary or notes from the audit to the Laboratory Technical Manager or QAO as part of the audit report. The summary identifies discrepancies found during the audit. Technical personnel are responsible for the inspection and monitoring of in-process and final data. Personnel independent of those having direct responsibility for the work performed audit the quality system and processes.

Representatives sent by customers and government or accrediting agencies often perform external audits. These audits are most often announced inspections, but sometimes are not announced. The Quality Assurance Officer, Laboratory Technical Manager or assigned deputy, and/or appropriate Department Manager accompany the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Technical Manager on any outstanding audit findings.

5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by customers and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past performance of an external performance audit.

5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP 1736. The corrective action program at Alpha Analytical uses the Nonconformance workflow in Qualtrax to document and follow through the corrective action/preventative action process for three main areas: nonconformance's within the laboratory, customer complaints and failed PT studies. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Nonconformance reports are tracked for closure date and the type. Reports to management include the listing of open nonconformance reports and the frequency of the type of nonconformance occurring. A QA designee monitors the completeness of the forms, as well as verifies the actions are complete and acceptable.

Customers will be notified within 5 days of any question(s) regarding validity of results.

5.7 Managerial Review

The management review occurs at least once per year as part of the strategic planning process. Documentation of the management review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, closure status, audit progress and other quality assurance actions. Meetings include discussion and progress on quality system initiatives since the last meeting.

Prior to the meeting, an agenda is distributed to all personnel expected to be in attendance. The meeting is chaired by the President. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method. Deviations from the existing SOP are allowed only upon approval of the deviation by the department manager and Quality Assurance Officer. This documentation must be either in form of written notice or email.

Alpha Analytical uses quality control samples to evaluate the following:

1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
2. Adequate tests to define the variability and/or reproducibility of laboratory results;
3. Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
4. Measures to evaluate test performance, such as detection limits and quantitation limits or range of applicability such as linearity;
5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
6. Selection and use of reagents and standards of appropriate quality;

7. Measures to assure the selectivity of the test for its intended purpose;
8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

   Note: All quality control samples are treated in the same manner as field samples.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance limits are used to determine the usability of the data. Control charts and/or calculated control limits monitor the long-term method performance by analyte, by instrument for water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional performance measures to ensure data comparability. Control limits are recalculated when trends are observed.

Where no reference method or regulatory criteria exist, the laboratory specifies the acceptance/rejection criteria in the SOP. The test SOP specifies the QC samples performed per batch of samples. The quality control samples are categorized into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Control Sample (LCS)
- Laboratory Control Sample Duplicate (LCSD)
- Matrix Spike (MS)
- Matrix Spike Duplicate (MSD)

Selection of samples for Duplicate, Matrix Spike (MS) & Matrix Spike Duplicate (MSD)

2. Duplicate samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

3. Matrix Spike (MS) / Matrix Spike Duplicate (MSD) samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be selected and analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.
   c. If MS/MSD is not required, LCS/LCSD may be substituted for precision and accuracy evaluation.

The frequency is dependent on the reference method and test protocol. The following is the default requirement for quality control checks in lieu of any other guidance. The frequency for each quality control sample is generally one (1) per every 20 samples.

5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. The analyst and reviewer sign and date the hardcopy of all manual integration. The analyst notes the rationale for performing the manual integration on the hardcopy printout and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \%R_{LCS}$$

where: *MV* = Measured Value
TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%R_{MS}$$

where: *MV* = Measured Value
TV = True Value
SV = Amount found in sample

Average ($\overline{X}$)

$$\sum_{i-1}^{n} X_i \Big/ n = \overline{X}$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{(R_1 + R_2)/2} * 100 = \%RPD$$

where: R_1 = Larger of two observed values
R_2 = Smaller of two observed values

% Difference (%D)

$$\frac{X - \overline{X}}{\overline{X}} * 100 = \%D$$

where: $\overline{X}$ = Average of all values
X = Result of measurement

Standard Deviation of the sample (S_x)

$$\sqrt{\frac{\sum \left(X - \overline{X}\right)^2}{n-1}} = S_x$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Standard Deviation (%RSD)

$$\frac{S_x}{\overline{X}} * 100 = \%RSD$$

where: $\overline{X}$ = Average of all values
Sx = Standard Deviation (n - 1)

Range of Logs (for microbiological enumeration analysis)

10% of routine samples are analyzed in duplicate and the range of logs is determined.

MDL (See 40CFR Part 136 for details)

where: *MDL* = The method detection limit

$$\left[\sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2 \Big/ n}{n-1}}\right] * t_{0.99} = MDL$$

X = Result of each measurement
n = Number of values
t(n-1,1 = .99) = The students' T value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Reporting Limit (RL)

Lowest calibration standard or greater

Control Limits

Upper Control Limit: $\overline{X} + 3 * S_x = UCL$
Lower Control Limit: $\overline{X} - 3 * S_x = LCL$

Warning Limits

Upper Warning Limit: $\overline{X} + 2 * S_x = UWL$
Lower Warning Limit: $\overline{X} - 2 * S_x = UWL$

Method of Standard Additions (MSA): (See EPA 7000A for details)

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard analyte solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration Cx is calculated:

$$C_x = \frac{SB\ V_S\ C_s}{(SA - SB)\ V_X}$$

where SA and SB are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_s should be chosen so that SA is roughly twice SB on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

5.10 Document Control

The Document Control Procedure (SOP/1729) describes the process for controlled and uncontrolled documents. The use of the revision number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is present on each page of the document. A master list of documents includes the unique identification. Each controlled copy includes the revision number, published date and page number.

Full document control includes the status of each document: active, inactive or superseded/archived. Inactive documents are procedures not currently requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and any subsequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and approval. The history section of the document in Qualtrax includes a description of the nature of the document change.

Standard Operating Procedures (SOPs) are instructions for repetitive or standard operations performed by the laboratory. The SOP author is the person familiar with the topic. The standard format for writing SOPs is set-up as a template for administration and technical SOPs. Each SOP is peer reviewed, authorized by management, and QA before final publication and implementation. Authorized signatories for controlled documentation include one or more of the following personnel: Company President, Quality Assurance Officer, Laboratory Technical Manager, Department Manager, Department Team Leader. Personnel acknowledge approved documents as read, understood and agreed to through electronic attestation forms associated with each document as SOP Attestation Tests which reside in Qualtrax.

SOPs must receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOP must conform to applicable requirements of analytical methods and certification agencies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or add methods, the scope of the existing method is extended, or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information and a new revision number is assigned and published in Qualtrax.

The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, customer instructions, Laboratory Work Instructions, data sheets, check lists and forms.

5.11 Detection Limits

Detection Limits (DLs), previously referred to as Method Detection Limits (MDLs), are determined for all analytes as specified in the NELAC TNI 2009 standards. DLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, Reporting Limits (RLs), are established. The RL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Laboratory reporting limits lie within the calibration range, at or above the RL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting limits are required below the lower level of the calibration curve, RL, or low-level check standard, method modifications are required. Refer to DL/LOD/LOQ SOP/1732. Note: ”J” Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Detection Limit (DL) with a “J” qualifier as long as there is a LOD study on file.

5.12 LOD/LOQ Studies

A. LOD (Limit of Detection) Verification

1. LOD (Limit of Detection) verification is required annually for each target analyte in which test results are to be reported below the lowest calibration standard (“J” values) for each instrument, matrix and prep procedure.

2. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

3. The validity of the LOD shall be confirmed by ***qualitative*** identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests, and >1 up to 4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

4. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

B. LOQ (Limit of Quantitation) Verification

1. LOQ (Limit of Quantitation) verification is required annually for each target analyte that is not reported below the lowest calibration standard for each matrix and prep procedure. LOQ is not required if an annual LOD verification is performed.
2. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the

claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria for accuracy.

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

The LOQ acceptance criteria are based on the established acceptance criteria for Laboratory Control Samples.

Refer to DL/LOD/LOQ SOP/1732

5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:

   a. Perform duplicate analyses on first 15 positive samples.

   b. Record duplicate analyses as D1 and D2 and calculate the logarithm of each result.

   c. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.

   d. Calculate the range (R) for each pair of transformed duplicates as the mean of these ranges.

6 Personnel

6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, customer specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

The H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During initial training, management provides access to documented operations procedures, observes personnel performance, and evaluates personnel proficiency. Management documents technical laboratory staff's proficiency initially and on a continuing basis through use of laboratory control samples and purchased proficiency evaluation standards.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and attestation process.

Either the Quality Assurance Officer, Operations Director and/or Technical Managers have the authority to stop work due to non-conformances and have the authority to resume work after it has been stopped.

6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Operations Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/1565 details the process for completing requirements and training to ensure personnel have adequate skills and competence for the job function. Initial training includes ethics training, Qualtrax Training, QA Basics, IT/LIMs including computer security.

A job description details the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of new employees and contract staff includes laboratory ethics and quality policies, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, report data values, that are not actual values obtained or improperly manipulated, or intentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section K of the Employee Handbook. Each employee must report personally or anonymously to the Laboratory Technical Manager, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics and ethics training occurs annually with all personnel.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix F)
- Initial and annual ethics training
- Internal audits conducted annually
- Adherence to Manual Integration SOP/1731

- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director
- Anonymous reporting to HR Director - This is accomplished by writing a detailed description of the suspected ethics breach and submitting the information, anonymously, to the Human Resource Director.
- "No-fault" policy encouraging reporting of incidences without fear of retribution
- Electronic tracking and audit trails through LIMs and instruments enable where available.

6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as-needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial employment. Orientation training includes laboratory business practices, employment specifications, Ethics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the job function.

Managers ensure the training for new employees and review the continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of higher learning.

Training of new personnel to any job assignment takes place on-site according to the Training Program procedure. Laboratory personnel may perform their assigned methods/protocols without supervision only after documentation of acceptable proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SOPs. Health and Safety training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Chemical Hygiene Plan and Material Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to the laboratory operation. All training is documented via the Training Attestation Form, which is signed by all in attendance that they understood and will implement what was presented to them.

Training is an on-going opportunity to evaluate the laboratory operations. The updating of SOPs, Quality Systems Manual and other related information documents all changes to the quality system. Training is documented via the Training Attestation Form or in Qualtrax with training test records.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee's responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual's training record.

6.4 Records

The QA Department is responsible for maintaining training records. Certificates, demonstration of capability forms and other records of training are placed in the individual's training file.

Appropriate personnel are notified through email and/or Qualtrax or by the QA department when a revision is complete for the controlled version of a document. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet for each of the Westboro and Mansfield Facilities

The laboratory functional areas include:

Administration and offices
Sample receiving
Sample management
Air analysis (Mansfield Facility only)
Microbiological (Westboro Facility only)
General analytical chemistry
Metals sample preparation
Organic sample preparation
Metals analysis
Volatiles gas chromatography (GC)
Volatiles gas chromatography/mass spectrometry (GC/MS)
Volatiles air analysis (Mansfield Facility only)
Semivolatiles gas chromatography/mass spectrometry (GC/MS)
Semivolatiles gas chromatography (GC)
Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropriate cabinets and properly disposed of as required. All flammable solvents are stored in OSHA and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste areas houses the sample and chemical waste before pickup by a licensed waste hauler.

7.1 Environment

Lighting, noise, humidity, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analytical equipment. Air-handling systems minimize airborne contaminants that may jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature and recorded in the data logger system. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/1728 and Mansfield SOP/1797)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and uncluttered work areas. The job descriptions list specific housekeeping duties. Records, samples and waste materials are the common cause for clutter in the laboratory.

. Removal of administration and laboratory records to the record storage area occurs to reduce clutter and ensure traceability. The individual filling the laboratory record box, labels the box with a number, the contents, date and laboratory area. Authorized personnel assign and record into a permanent record the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Boxes are stored onsite and off-site for the record retention period identified in the NELAC and EPA regulations, whichever is more stringent.

Sample management personnel remove samples to the sample storage area after all data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. The archive sample storage area is not controlled or monitored. Based on customer specifications, samples are properly disposed or returned to the customer.

Waste materials, expired reagents, expired standards and materials are disposed of and not stored in the laboratory. Hazardous waste labeled accumulation containers in the laboratory collect designated waste streams for later bulk disposal. Laboratory personnel remove the less than five-gallon accumulation containers when full from the laboratory and place the containers in the bulk hazardous waste area. Refer to the Waste Management and Disposal SOPS for Westboro: SOP/1728 and Mansfield SOP/1797. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, customers, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offices have the emergency contact list for the laboratory.

8 Equipment and Reference Materials

8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for most major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

A brief summary of some common preventive maintenance procedures is provided in Appendix E. All instrument preventative and corrective maintenance is recorded in the maintenance logbook assigned to the equipment. After maintenance or repair, the instrument must successfully calibrate following the method SOP. Laboratory personnel must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and consumables for analytical equipment. Backup instrumentation for some analytical equipment is available on site for use in case of major equipment failure. The person discovering or suspecting an equipment maintenance problem or failure tags the equipment with 'out of service' tag. If routine maintenance measures do not eliminate the problem, the Laboratory Technical Manager or Operations Director is notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and traceable maintenance logbooks in which to document manufacturer's recommended maintenance procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetitive or on-going equipment problems other than normal maintenance requirements on nonconformance action forms. The nonconformance action form notifies management and the Quality Assurance Officer of a problem affecting the performance and data quality.

The laboratory groups some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

- Equipment name;
- Manufacturer's name, type identification, serial number or other unique identification;
- Date received, date put into service, condition when received;
- Current location;
- Details of past maintenance and future schedule;
- A history of any damage, malfunction, modification or repair;
- Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing bromthymol blue (BTB) on each day of media preparation.

8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment location, serial number, model number, and purchase date. The unique identification number is attached to the piece of equipment.

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

8.3 Laboratory Water

Laboratory water is purified from central DI water systems and piped to all laboratory areas. In Westboro, the QA Department samples the laboratory grade water and submits the samples for analysis by the lab to document the water meets the drinking water certification criteria. The Laboratory Water Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/1738). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

TABLE 8-1

Parameter	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon (Westboro only)	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia Nitrogen (Westboro only)	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn (Westboro only)	Monthly (Required Annually)	< 0.05 mg/L
Total Metals (Westboro only)	Monthly (Required Annually)	< 0.1 mg/L

Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL
Water Quality Test (Biosuitability) (Westboro only)	Annually	0.8 – 3.0 ratio

8.4 Reference Materials

Reference materials include: Class 1 weights, NIST thermometers and reference standards. Logbooks record the reference materials used for calibration and verification. The Department Manager or QA Department maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area records the unique identifier on the reference standard certificate and the Department Manager maintains the certificate. The identifier allows traceability from the certificate to the analytical data.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

9.2 Traceability of Calibration

The program of calibration and/or verification and validation of equipment is such that measurements are traceable to national standards, where available. Calibration certificates indicate the traceability to national standards, provide the results, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. A body that provides traceability to a national standard calibrates reference standards. The laboratory maintains a permanent file of all such certifications.

9.3 Reference Standards and Materials

Alpha Analytical has a program for calibration and verification of reference standards. The results and program are recorded in the appropriate instrument logbook. Required in-service checks between calibrations and verifications are described in method SOPs and are recorded in the appropriate instrument logbook.

Calibration standards are maintained within the area of consumption. A logbook of use is maintained and use is limited strictly to method required calibrations. Each calibration standard is identified as to test method used, date received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are stated in applicable test method SOPs.

Preparation of standards must be performed using Class A glassware. Class A glassware must be used for all processes involving quantitative analyses.

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and for no other purpose.

Standards and reagents are uniquely identified as outlined in Westboro SOP 1745 and Mansfield SOP 1816.

9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves. Calibrations are tagged to the specific instrument through use of the instrument logbook and or sequence file documentation.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and reference method specifications. Before the start of each analytical sequence, initial calibration is

verified by using a continuing calibration standard. Calibration verification or continuing calibration uses the same standard as the ICAL unless method specifies otherwise. The ICV is from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, decafluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by analyzing a set of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each solution is analyzed once, unless the method or the customer requires multiple analyses. The relative response factor for each analyte is calculated for internal standard calibration. The calibration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within method criteria.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on

the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent drift (%d) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percent differences or percent drift for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed until the continuing calibration analysis meets all criteria, or a new calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.3 Cold Vapor Atomic Absorption Spectrophotometry (CVAA)

An initial calibration is performed daily with freshly prepared working standards that bracket the expected concentration range of the sample. A minimum of a three-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified every 10 samples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within ± 10% of the true value.

9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value for EPA Method 200.7 and 10% for SW846 6010 methods. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-certified every year. Records of thermometer calibrations are retained by the QA Department. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

NOTE: Electronic-based thermometers are calibrated on an annual basis. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to be applied and the expiration of the calibration check. Certificates are kept on file in the QA Department.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage or expired calibration.

9.5.7 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights, which bracket the weight to be measured. Additional calibration check procedures are performed on balances utilized in Microbiology laboratory. This additional procedure consists of a deflection test, which is performed to ensure that 100mg is detectable at a weight of 150 grams.

The balance logbook lists the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

9.5.8 Mechanical volumetric pipettes

Delivery volumes for the mechanical volumetric pipettes (i.e. Eppendorf) are checked and recorded gravimetrically before use and on a quarterly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook. Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range for the analysis are used for calibration. A second source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST

certified. Standard buffer solutions are not retained or re-used. The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement to NIST.

9.5.11 Conductivity Meters

Three calibration standards of potassium chloride (KCL) solutions are analyzed annually on each instrument range. The calibration standards are used to verify instrument performance. The acceptance criteria are defined in the test SOP. If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is used to calibrate each range of the instrument. A second standard is used to check the calibration each day the meter is used. The check standard is near the measurement range for the samples to be analyzed. The acceptance criterion is ± 20% of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceable is used to allow traceability. The check standard is performed at the end of the analysis run or at least after every 20 samples.

9.5.12 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60°C for 24 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.

10 Test Methods and Standard Operating Procedures

10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or customer-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the customer.

The reference methods define the instrument operating conditions. In many of the reference methods, a range or general guidance on the operating conditions is defined. Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at the time of the analysis are documented in the appropriate laboratory or sequence log. A revision to the SOP takes place, when a day to day change in the operating condition improves performance for all matrices.

The laboratory SOPs include the operation of measurement equipment. The SOPs contain the - following information, as applicable:

- The equipment used in the procedure, including equipment type
- Equipment calibration and process for obtaining the measurement from the calibration
- The step by step instructions to perform the measurement
- Acceptance criteria for the calibrations
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- The basis used for the calibration standards such as traceability to NIST or EPA or demonstration of comparability
- Frequency at which the equipment will be calibrated, adjusted and checked
- The records maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in electronic form through Qualtrax. Each SOP clearly indicates the published date of the document and the revision number.

10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents in the Alpha Qualtrax system. Directories are available for each laboratory area and administrative area in appropriate subfolders. Each SOP includes or references where applicable:

1) identification of the test method and where applicable;
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application;
5) summary of method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies
10) reagents and standards
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measurements;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the customer and other recipients of the relevant reports.

The customer requests the reference method for sample analysis usually based on the regulatory program. The customer services staff may assist the customer with method selection when the customer specifies the regulatory program, but is unsure of the correct method required. The Laboratory Technical Manager or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on customer-defined method performance criteria. Customer services may recommend a procedure that meets the customer method performance criteria.

10.5 Method Validation/Initial Demonstration of Method Performance

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method.

10.6 Sample Aliquots

The aliquot sampling process from a submitted sample is part of a test method. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Personnel record observations made during aliquot sampling in the test method logbooks.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks. A second person recalculates all manual calculations. An independent qualified analyst also reviews the data. A Customer Services representative reviews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Customer. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Customer within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Customer Services Department of the sample number(s), Customer name, analysis and sample results (preliminary or confirmed). The Customer Services Department notifies the customer.

The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

a) The laboratory retains records of manufacturer’s statement of purity, of the origin, purity and traceability of all chemical and physical standards.
b) Original reagent containers are labeled with the date opened and the expiration date.
c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.

d) Where calibrations do not include the generation of a calibration curve, records show the calibration date and type of calibration standard used.
e) All prepared reagents and standards are uniquely identified and the contents are clearly identified with preparation date, concentration and preparer's initials. These procedures are outlined in Westboro SOP/1745 and Mansfield SOP/1816.

10.9 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

the software systems perform the required functions accurately,

the users understand how to use the system, and

auditors can assure themselves of the validity of the analytical data.

The computer systems used at Alpha Analytical are purchased. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Alpha Analytical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.
2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems.

Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/1562 and Computer Security SOP/1563. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Customers or Alpha's Couriers deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Customer service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are packed to minimize or prevent breakage. The containers are placed in plastic coolers or shipping packages and Chain-of Custody forms, seals (if requested) and labels enclosed. The bottle order is shipped by third party, picked up by the customer or customer representative or delivered by Alpha courier to the customer.

11.1 Sampling Supplies

11.1.1 Sample Containers

Sample containers provided by Alpha Analytical include labels, preservatives and a blank chain of custody form. Preservatives and containers are lot controlled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collection of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checks are performed by Microbiology staff and results recorded in Microbiology Sample Container Sterility Log.

Sample Containers for collecting Air samples (TO-15) are cleaned and prepared according to SOP 2190 "Cleaning and Preparation Procedures for Equipment used to collect Air sample for analytis of Volatile Organic Compounds".

11.1.2 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

11.1.3 Reagent Water

Alpha Analytical supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on customer specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/1738).

11.2 Sample Tracking

Alpha Analytical uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Information Management System (LIMS) to uniquely identify and track samples and analytical data throughout the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project if requested)
- Project name or description
- Analyses requested (per matrices received)
- Sample number (unique to this sample)
- Sample descriptions (customer ID, including number of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due

11.2.1 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

- Customer's name and address
- Notation of special handling instructions
- Additional comments or instruction for the laboratory
- Purchase order number(s), if applicable

Alpha Job Numbers (Process for assigning numbers)

Alpha Job Numbers are unique #'s automatically designated by our LIMS computer system for every individual customer project.

There are 3 parts to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

For example…. L0904165 ---- Year 2009 and 4,165th job to be logged in this year.

The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives its own bar-coded label. For example, L0904165-09A is the first container of the 9th sample listed on a customer's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The customer and customer services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediate notify customer services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. The problem is brought to the attention of a customer services manager who reports it to the customer. Plans for disposition of the affected samples or container are agreed upon with the customer, carried out, and recorded in the project records. Sample hold times and preservations are listed on the Alpha website (www.alphalab.com) under Support Services "Sampling Reference Guide".

11.4 Sample Receipt Protocols

The sample management staff receives all samples. A unique job number is assigned to each shipment of samples received from a customer. The in-house records for the incoming job, including the internal Chain-of -Custody, are initiated with a Sample Delivery Group (SDG) form. The customer, and Alpha courier and/or the sample management personnel sign the sample custody form at the time of receipt at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, SDG form and the sample custody form are completed for external courier delivered samples.

The sample management staff examines the shipping containers, their contents, and accompanying customer documentation. Information about the sample identification, the location, date and time of collection, collector's name, preservation type, sample type, presence and condition of custody seals, the state of preservation of the samples and other required information is noted on the SDG form. Any discrepancies in documentation or problems with sample condition such as appropriate sample containers, thermal preservation variation, holding times and adequate sample volumes are noted and brought to the attention of the customer via the nonconformance action form, The login staff or project manager contacts the client via email or or by phone. The Customer Services Manager provides clarification or further instruction to the sample management staff on the processing of the samples that are incomplete or missing required information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, customer sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and customer directives. Refrigerators are monitored and controlled to be within 4 ± 2°C. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Customer Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or customer specific requirements are recorded on nonconformance action forms and contact made with the customer by the Customer Services Manager or designee.

Sample storage facilities are located within the sample management area or in designated sample storage areas within the analytical departments. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure. Samples are returned to the sample storage area after the sample portion is removed for analysis. Extracts and digestates are tracked and follow the same internal custody operation. Extracts and digestates are removed to the waste disposal area after analysis for proper disposal.

Sample storage precautions are used to ensure that cross contamination does not occur during sample storage. Refrigerator storage blanks are monitored bi-weekly for volatile compounds.. The storage blank information allows the assessment of potential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are recorded continuously in the data logger system. Corrective action is done as necessary when temperatures are not within the control criteria. In both the Westboro and Mansfield facilities, Automated Data loggers are linked to thermocouples in custody refrigerators and freezers in the Sample Storage areas as well as department standards/storage refrigerators and freezers. The Data logger is calibrated and certified by an outside vendor on a quarterly basis. Refrigerators and/or freezers not connected to the Data Logger system have temperatures measured with NIST traceable thermometers. Temperature records indicate the thermometer or sensor (Data logger) used for obtaining the measurement.

11.6 Sample Disposal

Samples are held for 21 calendar days after the report is released to the customer. Upon written customer request samples may be held longer in an uncontrolled area. Requests for controlled sample storage must be arranged at the time of contractual commitment. Air canister samples are held for 3 days after the report is released to the customer.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the customer, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP 1797 defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to customers. Samples in storage are retained a minimum of 21 calendar days after reporting the results to the customer. Any samples requiring more than 21 calendar days are archived. Air canister samples requiring storage more than 3 business days require prior approval.

When a customer has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any customer specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of this laboratory. Customers are informed that a commercial carrier will deliver their samples if required.

12 Records

Alpha Analytical has a record system that produces accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized customers, agencies or another regulator. Note: Ohio VAP requires notification before disposal of any VAP records.

12.1 Record Keeping System and Design

The record keeping system allows reconstruction of laboratory processes that produced the analytical data of the sample.

a) The records include the names of personnel involved in sampling, preparation, calibration or testing.

b) Information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.

c) The record keeping system provides retrieval of working files and archived records for inspection and verification purposes.

d) Documentation entries are signed or initialed by responsible staff.

e) Generated data requiring operator logging on appropriate logsheets or logbooks are recorded directly and legibly in permanent ink

f) Entries in records are not obliterated by any method. Corrections to errors are made by one line marked through the error. The person making the correction signs and dates the correction.

g) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

12.2 Records Management and Storage

1. Records including calibration and test equipment, certificates and reports are safely stored, held secure and in confidence to the customer.
2. The laboratory maintains hardware and software necessary for reconstruction of data.
3. Records that are stored or generated by computers have hard copy or write-protected backup copies.
4. Alpha Analytical has established a record management system, for control of hard copy laboratory notebooks.
5. Access to archived information is carefully controlled and is limited to authorized personnel. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.
6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or transferred according to the customer's instructions. A plan will be

developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

1. Analysts work sheets and data output records,
2. Reference to the specific method,
3. Calculation steps including definition of symbols to reduce observations to a reportable value,
4. Copies of all final reports
5. Archived SOPs,
6. Correspondence relating to laboratory activities for a specific project,
7. All nonconformance action reports, audits and audit responses,
8. Proficiency test results and raw data,
9. Data review and cross checking.

The basic information to tie together analysis and peripherals such as strip charts, printouts, computer files, analytical notebooks and run logs for Alpha Analytical includes:

1. Unique ID code for each Laboratory sample or QC sample;
2. Date of analysis;
3. Instrument identification and operating conditions;
4. SOP reference and version;
5. Calculations;
6. Analyst or operator's initials/signature.

In addition, Alpha Analytical maintains records of:

1. Personnel qualifications, experience and training
2. Initial and continuing demonstration of proficiency for each analyst
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records. Use of electronic signatures has been approved by regulatory agencies.

12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to records pertaining to:

a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes inter-laboratory transfers of samples, extracts and digestates.

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) Automated sample handling systems;

n) Records storage and retention; and

o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.

p) The COC records account for all time periods associated with the samples.

q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure.

r) Common carrier documents.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the customer and in accordance with relevant environmental regulations.

Customers specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Customers specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival. Affidavits are required with each report or series of reports generated for a particular project for Ohio VAP reports.

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report format. The standard report format includes:

1. Title: “Certification of Analysis”
2. Name and address of the laboratory
3. Laboratory Job Number, page number and total number of pages included in the report.
4. Name and address of the customer
5. Alpha sample number, Customer identification, Sample location
6. Samples identified that do not meet the sample acceptance requirements for project.
7. Date of sample receipt, sample collection, analysis date and time, report date and analyst
8. Identification of data reported by subcontractors
9. Test name and EPA reference method number
10. Delivery method and sampling procedures when collected by lab personnel
11. Deviations or modifications that affect data quality and/or data integrity. These deviations or modifications are included in narrative statements and/or data merger files.
12. Statement that results relate only to the sample tested
13. Statement that report must be copied in full unless the laboratory provides written permission for partial copies
14. Glossary, References and limits of liability
15. Units of measure and reporting detection limit
16. Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
17. Signature, title and date of report

18. A "Certificate/Approval Program Summary" page is included at the end of the report that identifies analytes for which Alpha Analytical holds certification and for those analytes reported that it does not. This summary also includes the certification numbers for either NELAP certified states, State certifications (e.g. Massachusetts laboratory certification identification number)..
19. Alpha Analytical does not accept samples from private residents for drinking water analysis and therefore maximum contaminant levels are not necessary. If Alpha were to change its policy and report drinking water samples, MCLs would be included with the report.

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory's expense.

The laboratory notifies the customer in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature. The customer will be notified prior to changes in LIMs software or hardware configurations that will adversely affect customer electronic data.

13.1 Data Qualifiers

The following data qualifiers are used in conjunction with analytical results depending on the definition, state or regulatory program and report type.

Note: "J" Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Method Detection Limit (DL) with a "J" qualifier as long as there is a LOD study on file. (See section 5.11)

Data Qualifier	Qualifier Information	Regulatory Requirement
A	Spectra identified as "Aldol Condensation Product".	CT RCP, NC
B	The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at <5x the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than 10x the concentration found in the blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone)	EPA Functional Guidelines 'MassDEP MCP, CT RCP, NJ-TO15/LL-TO15; NJ Tech Guidance 2014
C	Co-elution: target analyte co-elutes with a known lab standard (i.e. surrogates, internal standards, etc.) for co-extracted analyses.	
D	Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.	NJ-TO15/LL-TO15 - Air only EPA Functional Guidelines; EPA Region 2,5
DL	Same was re-analyzed at a dilution. Qualifier applied to sample number.	
E	Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.	EPA Region 2,5 CT RCP, NJ-TO15/LL-TO15

G		The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.	In-house/Forensics.
H		The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.	NELAC
I		The lower value for the two columns has been reported due to obvious interference.	In-house.
J		Estimated value. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs),
JN (NJ)		Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.	EPA Functional Guidelines 'NJ-TO15-LL
ND	DU-J	Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for same-related analysis	In-house
P	All DU	The RPD between the results for the two columns exceeds the method-specified criteria.	MassDEP MCP, CT RCP
Q	All DU	The quality control sample exceeds the associated acceptance criteria. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)	
R	All DU	Analytical results are from sample re-analysis	Customer-specific
RE	All DU	Analytical results are from sample re-extraction.	Customer-specific
S		Analytical results are from modified screening analysis	

13.2 Compound Summation for Organic Analyses

In order to be compliant with regulations from certain states, Alpha Analytical has created the following Summation Rules to cover reporting "Total Analytes". The following are an example of several compounds that can be reported as "Totals":

Volatiles:	
1,3-Dichloropropene, Total	cis + trans isomers
Xylenes, Total	m/p + o isomers
1,2-Dichloroethene, Total	cis + trans isomers
Trihalomethanes, Total	Chloroform + Bromoform +
	Dibromochloromethane +
	Dichlorobromomethane
PCBs:	
PCBs, Total	Sum of reportable Aroclors
	(all Aroclors reported for the project)

The following are the summation rules that the LIMs uses to calculate the Total values:

Summation Rules:		
H + H = H		Key:
H + J = J		H = Hit (above RL)
J + J = J		J = J-flagged value
H + ND = H		ND = U-flagged value
J + ND = J		
ND + ND = ND		

The ND values are considered "0" during the calculations.
The "E" flagged values (over the calibration) are ignored and not utilized during the calculations.
Any "N" flagged values (do not report) are ignored and not utilized during the calculations.
For dual-column analysis, the Total is reported as part of column "A" data, unless all individuals are reported from "B" column.

For analytical group summations, the Total is reported based on the associated "Reporting List". For example, if only 7 Aroclors are requested, then the Total is based on 7 Aroclors, not 9.

The RL and MDL for Totals will always be the lowest of the individual compounds used in the summation.

For each Total summation, two values are calculated: TOTALH (calculated from all associated hits above the R L– used in DU reporting formats) and TOTALJ (calculated from all associated hits and J flagged values – used in DJQL reporting formats). Total concentrations are calculated for all samples and QC samples (however, recoveries are not calculated since they are only calculated for the compounds spiked)

If a Total summation is requested, the individual compounds must also be reported.

14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests. Differences between Request/Tender and Contracts must be resolved before work commences.

The Purchasing SOP/1726 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The Purchasing SOP/1726 describes the details for receipt and inspection of purchased product.

The Purchasing SOP describes the process for raising, review and placement of purchase orders. It is company policy to purchase from third party certified suppliers and subcontractors wherever possible. Purchases must be from suppliers approved by the Laboratory. Laboratory or sampling subcontractors specified by the customer are noted as “Trial” on the purchase order. This identifies the subcontractor as a non-approved subcontractor.

The laboratory maintains list of approved vendors (Form 13-01) and subcontractors from whom it obtains support services or supplies required for tests.

14.1 Subcontracting Analytical Samples

Customers are advised, verbally and/or in writing, if any analyses will be subcontracted to another laboratory. Any testing covered under NELAC that requires subcontracting, will be subcontracted to another NELAC accredited laboratory for the tests to be performed. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties’ certifications or approvals. This document must indicate current approval for the subcontracted work. Any sample(s) needing special reports (*i.e.*, MCL exceedance) will be identified on the chain of custody when the laboratory subcontracts with another laboratory. Subcontractor Laboratory Certifications are located in Qualtrax under Customer Services folder

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. The quotation form lists the subcontractor in order to notify the customer of any subcontracted work. Customer notification of subcontracted work is in writing before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The person responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor.

15 Customer Relations

15.1 Customer Service

The majority of the customer services occur from personnel in the administration, sample receiving and sampling areas. Customer service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with customers must document and review customer specific project requirements. Call Tracker is used to document communications with customers (SOP/1723). Personnel must document customer interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and customer requests following the laboratory defined procedures.

15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Technical Manager address all capacity and capability issues. Where conflicts in workload arise, customer notification is immediate. The Project Communication Form (PCF) contains the documentation of all project information. Cooperation between laboratory and customer services staff allows direct communication and scheduling. Management arranges complex scheduling and coordination between departmental areas.

15.3 Complaint Processing

The laboratory staff documents all customers or other parties' complaints or concerns regarding the data quality or laboratory operations. The Nonconformance Report records complaints, correcting the concern, and resolving the concern with the customer or other party. The process uses the same form and process as the nonconformance action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiry and Complaint SOP/1722 is immediate to ensure the corrective action has effectively solved the concern.

16 Appendix A – Definitions/References

The following definitions are from Section 3.0 of the 2009 TNI Standard. The laboratory adopts these definitions for all work performed in the laboratory.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. (TNI)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (TNI)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (EPA QAD glossary)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation. (TNI)

Assessment (Clarification): The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria.

Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI).

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A

preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (TNI)

Blanks include:

Equipment Blank: a sample of analyte-free media, which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

Instrument Blank: a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) 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 procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses, (TNI)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

Calibration: set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or

measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).

2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the Laboratory with a certificate of analysis or purity, or prepared by the Laboratory using support equipment that has been calibrated verified to meet specifications.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Method: A defined technical procedure for performing a calibration.

Calibration Standard: A substance or reference material used to calibrate an instrument. (TNI)

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols (TNI)

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq*., Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures (TNI)

Customer: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

Continuing calibration verification: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Completeness: the percentage of measurements judged to be valid compared to the total number of measurements made for a specific sample matrix and analysis.

Data Quality Objectives (DQO):

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (TNI)

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: (previously referred to as Method Detection Limit –MDL) the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.

Detection Limit (DL) (Clarification): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq*., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C 1251 et seq., Public Law 92-50086 Stat. 8.16, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field Measurement: The determination of physical, biological, or radiological properties, or chemical constituents; that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation. (TNI)

Finding: an assessment conclusion, referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. (TNI)

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

Holding Times: The maximum time that can elapse between two (2) specified activities. (TNI)

The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified

requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (TNI)

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory: Body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (TNI).

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the customer. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory. (TNI)

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

Limit of Detection (Clarification): 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%.

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence. (TNI)

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias.

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work. (ANSI/ASQ E4-2004)

Management System: System to establish policy and objectives and to achieve

those objectives (ISO 9000).

Matrix: The substrate of a test sample. (TNI)

Matrix Spike (spiked sample, fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (TNI).

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (TNI).

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). (TNI)

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed. (TNI)

Method Detection Limit: (now referred to as Detection Limit) one way to establish a Detection Limit, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Method Detection Limit (MDL) (Clarification): The MDL is one way to establish a Detection Limit, not a Limit of Detection.

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Mobile Laboratory: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel. (TNI)

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute. (NMI). (TNI)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which TNI is a part.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI).

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Procedure: A specified way to carry out an activity or a process. Procedures can be documented or not. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (TNI).

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is the type and quality needed and expected by the customer. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements or quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users. (TNI)

Quality Manual Clarification: Alpha Analytical refers to Quality Manual as Corporate Quality Systems Manual (CQSM). (Alpha)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities. (TNI)

Quality System Matrix: These matrix definitions are to be used for purposes of batch and quality control requirements: (TNI)

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location. (TNI)

Representativeness: the degree to which the sample represents the properties of the particular sample being analyzed.

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure. (TNI)**Second source calibration verification (ICV):** A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent. (TNI)

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (TNI)

Standard Method: a test method issued by an organization generally recognized as competent to do so.

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Technology: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques. (TNI)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Tentatively Identified Compound (TIC): A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations. Tentatively Identified Compounds, if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported.

Test Method: An adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e. the air, water and land) upon which human life depends. (US-EPA)

Validation: the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring

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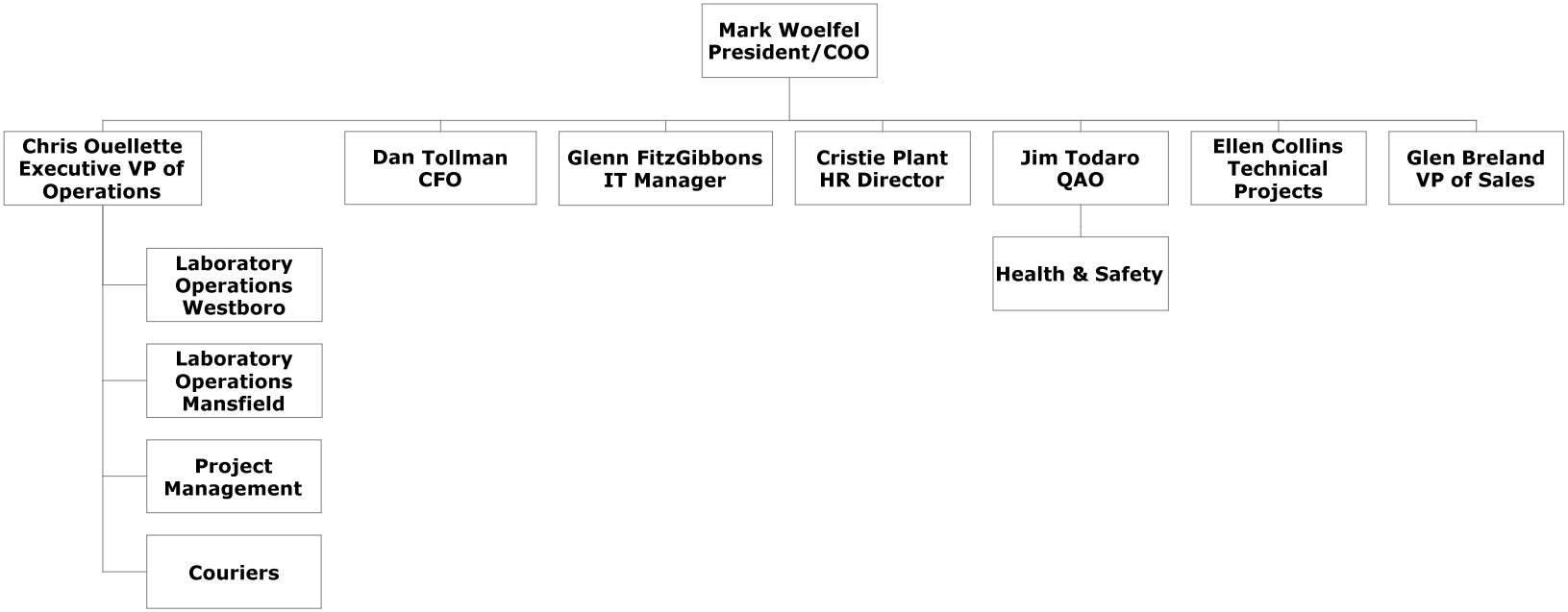
17 Appendix B – Organization Charts

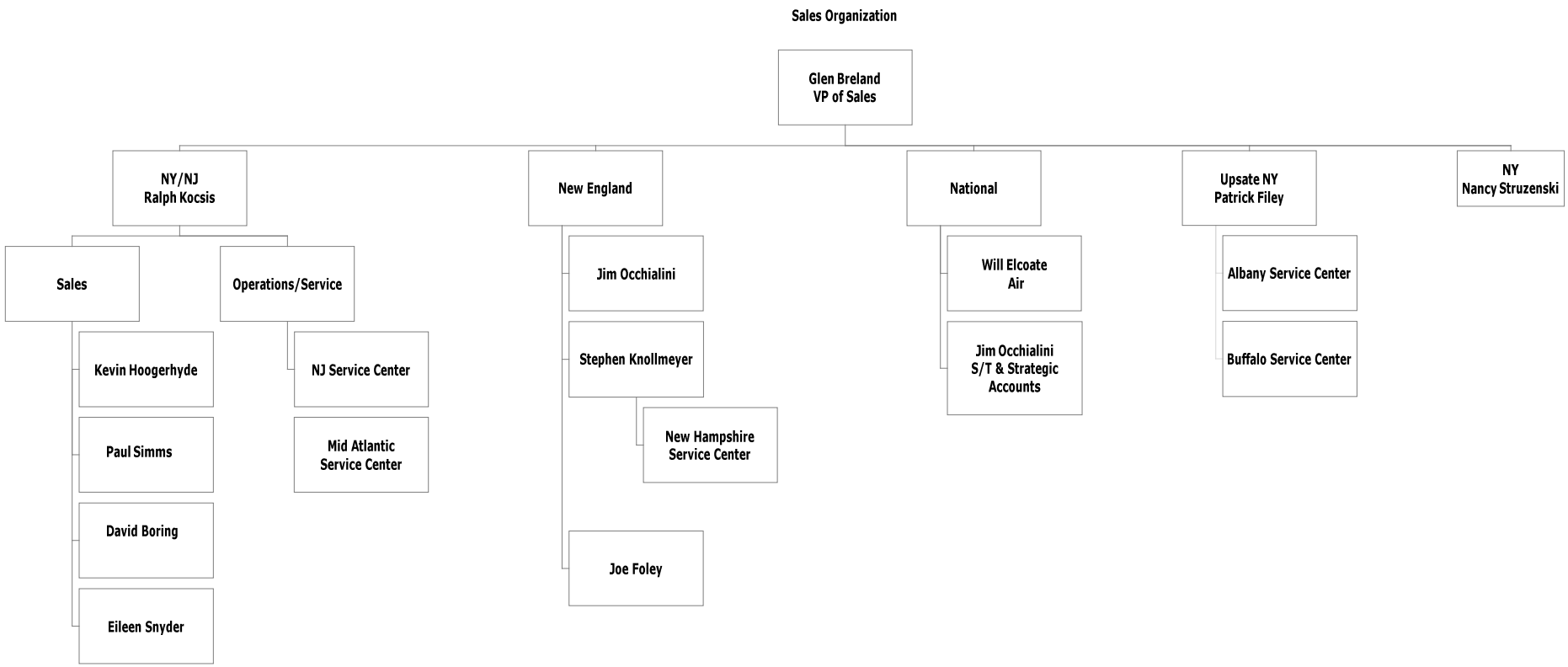
The following charts provide an overview of the organizational structure of Alpha Analytical. The chart also identifies the key personnel responsible for the listed positions. For the various laboratory areas, the individual departmental supervisors are noted. For a listing of all current key personnel, please refer to Section 18, Appendix C.

Updated 10/4/2014

2014
Alpha Analytical
Company Organizational Chart

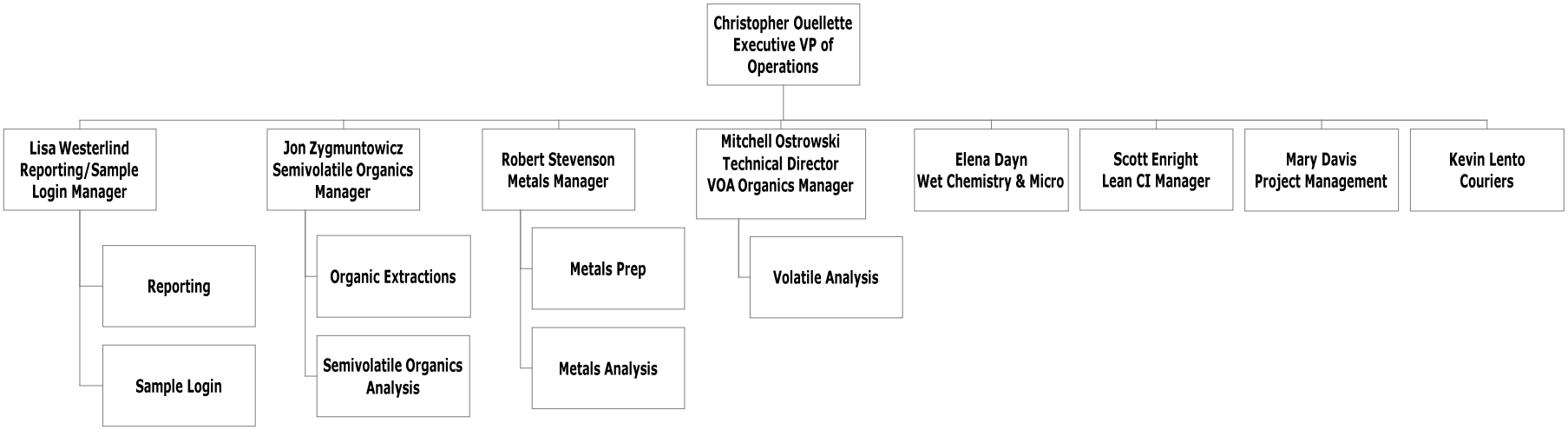


Updated 08/11/2015



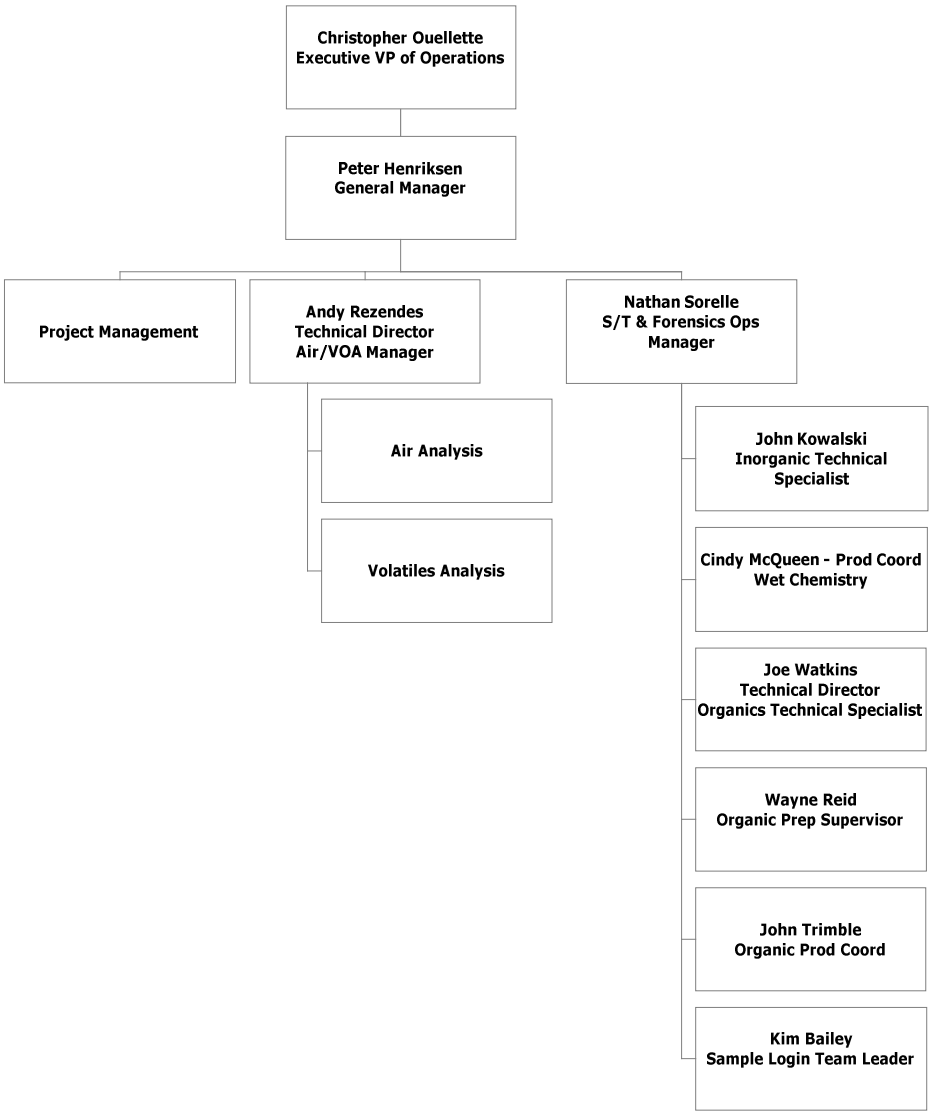
Updated 10/4/2014

Alpha Analytical
Laboratory Organizational Chart
WESTBOROUGH



Updated
08/11/2015

Alpha Analytical
Laboratory Organizational Chart
MANSFIELD



18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 10/7/2014.**

President / COO: Mark Woelfel
Executive VP of Operations: Christopher Ouellette
CFO: Dan Tollman
Laboratory Technical Manager - Westboro: Mitchell Ostrowski
Laboratory Technical Manager - Mansfield: Joseph Watkins
Laboratory Technical Manager- Air, Volatiles Manager - Mansfield: Andy Rezendes
Quality Assurance Officer/Health & Safety Manager: James C. Todaro
VP, Technical Projects: Ellen Collins
VP Technical Sales/Sales Manager: Glen Breland
VP, Technical Sales: James Occhialini, Ralph Kocsis, Pat Filey, Kevin Hoogerhyde, Steven Knollmeyer
Technical Sales Reps: Paul Simms; Joe Foley, David Boring
General Manager, Mansfield: Peter Henriksen
Director of Project Management: Mary Davis
National Air Account Manager: Will Elcoate
Information Technology Manager: Glenn Fitzgibbons
Human Resources Director: Cristie Plant
Health & Safety Officer: Jay Troy
Forensic & S/T Operations Manager, Mansfield: Nathan Sorelle
SVOA/Extractions Manager, Westboro: John Zygmuntowicz
VOA Department Manager, Westboro: Mitch Ostrowski
Wet Chemistry Department Manager, Westboro: Elena Dayn
Metals Department Manager, Westboro: Robert Stevenson
Login Manager/ Reporting Manager, Westboro Lisa Westerlind
Quality Systems Specialists: Amy Rice, Rene Bennett, Jason Hebert, Blake Buckalew
Purchasing: Rosemarie Pederson
Logistics Manager: Kevin Lento
Equipment Specialists: Pat Sullivan, Greg Yogis

19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals		
Equipment	**Frequency**	**Type of Calibration or Maintenance**
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights
COD Reactor	annually annually	complete operations check by service technician (external) reaction temperature verification
Conductivity Bridge	annually each use	verification of cell constant complete operations check by service technician (external) calibration verification
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check
DO Meter	annually each use	complete operations check by service technician (external) calibration against air as specified by manufacturer
Emergency/Safety Equipment	annually monthly	fire extinguishers and emergency exit lighting check eye washes, showers, fire blanket and first aid kits checked
Freezers	daily	temperature verification
Gas Chromatographs	as needed as needed beginning and end of batch and 10 to 20 samples as per method	injection port preparation; cleaning of detectors initial multi-point calibration continuing calibration verification (CCV) against initial calibration
ICP	Every other day Daily Annually Annually As needed	Change pump tubing Calibration, profile Complete operations check by service technician (external), Linear Dynamic Range determination Clean torch, clean nebulizer, clean spray chamber
Lachat analyzer	Daily As needed	Calibration, clean lines Change tubing, change O-rings
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or daily	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV
Mercury Analyzer	monthly each use	clean cell and change pump windings calibration using multi-point curve
Auto-pipettes	Monthly Annually	verification of accuracy verification of precision
Microwave	Quarterly Annually	power and temperature verification RPM verification
Ovens	annually daily	complete operations check by service technician (external) temperature verification
pH Meters	annually each use	complete operations check by service technician (external) calibration using certified buffers
Refrigerators (General Use)	daily	temperature verification
Refrigerators (Sample Management)	daily	temperature verification
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration
TCLP Rotator	annually	RPM verification
Thermometers (Mercury/Alcohol)	annually	calibration against NIST traceable thermometer (internal)
Thermometers (digital)	Quarterly	calibration against NIST traceable thermometer (external)
Thermometer (NIST Traceable)	annually	calibration and certification of conformance (external)
Turbidity meter	annually each use	cleaning & operations check by service technician (external) calibration using formazin
Weights (Class S-1)	annually	service/calibration and certification of conformance (external)

20 Appendix E – Alpha Code of Ethics Agreement

Alpha Analytical, Inc.
Ethical Conduct and Data Integrity Agreement

A. ***Personal Pledge:*** I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report data, test results and conclusions that are accurate, precise and of the highest quality.

B. ***Protocol Pledges:*** I agree to adhere to the following protocols and principles of ethical conduct in fulfilling my work assignments at Alpha:

1. All work assigned to me will be performed using Standard Operating Procedures (SOPs) that are based on EPA approved methods or Alpha methods.

2. I will only report results or data that match the actual results observed or measured.

3. I will not intentionally nor improperly manipulate or falsify data in any manner, including both sample and QC data. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented in the appropriate laboratory notebooks and raw data and include my initials or signature and date.

4. I will not intentionally report dates and times of analyses that are not the actual dates and times the analyses were conducted.

5. I will not intentionally represent another individual's work as my own or represent my work as someone else's.

6. I will not make false statements to, or seek to otherwise deceive Alpha staff, leaders or customers. I will not, through acts of commission, omission, erasure or destruction, improperly report measurements, standards results, data, test results or conclusions.

C. ***Guardian Pledge:***

1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Technical Manager or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.

2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha's QA Officer, senior leadership or corporate officers, including the President of the company.

3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or improper. Also, I will not discourage, intimidate or inhibit a staff member who may

choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. ***Agreement Signature:*** I have read and fully understand all provisions of the *Alpha Analytical Ethical Conduct and Data Integrity Agreement*. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

_____
Employee Signature

_____
Printed Name

_____
Date

Review Requirements
The *Ethical Conduct and Data Integrity Agreement* must be signed at the time of hire (or within 2 weeks of a staff member's receipt of this policy). Furthermore, each staff member will be required to review and sign this agreement every year. Such signature is a condition of continued employment at Alpha. Failure to comply with these requirements will result in immediate discharge from Alpha employment. This agreement is not an employment contract and does not modify in any manner the company's *Employment-at-Will* Agreement.

21 Appendix F – Floor Plan Westboro Facility

22 Appendix G– Floor Plan Mansfield Facility

23 Appendix H – Job Titles and Requirements

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Technical Manager (Director) Organic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 24 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of organic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Inorganic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of inorganic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Microbiology Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in the Biological Sciences, including at least one course having microbiology as a major component. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of microbiological analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Quality Assurance Officer	BS/BA in Chemistry, Biology, Environmental or related Science	Two (2) years Environmental Laboratory Experience	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Knowledgeable in Federal, State Programs (NELAC, etc.) 3. Able to develop QA/QC policies and certification requirements 4. Able to develop training programs for quality procedures 5. Documented training and/or experience in QA and QA procedures 6. Knowledge of safe laboratory practices and emergency protocols

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Laboratory Coordinator	High School Diploma; Associates or BS/BA in Chemistry, Biology or Environmental or related Science preferred	1 year +	1. Knowledge of safe laboratory practices and emergency protocols 2. Proficient in all methods and SOP's within their department 3. Experience with and understanding of LIMS 4. Proven ability to meet TAT (turnaround times)
Quality Systems Specialist	BS/BA Chemistry	2 years +	1. General knowledge of laboratory methods 2. Experience with and understanding of LIMS 3. Strong attention to detail 4. Strong oral/written communication and organizational skills 5. Knowledge of QA/QC policies and certification requirements
EH&S Coordinator	High School or Equivalent	2 years +	1. General knowledge of lab operations 2. Detailed knowledge of safe lab practices and emergency protocols 3. Hazardous Waste Management and RCRA Regulation Training 4. DOT Hazardous Materials Regulations Training 5. OSHA Compliance Training 6. Able to develop and deliver new hire and ongoing safety training programs
Lab Technician I	HS or Equivalent	0-1 years. 1+ years preferred.	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician II	HS or Equivalent	2-4 years	1. All skills of Lab Technician I 2. Trained in majority of technician skills relative to department
Lab Technician III	HS or Equivalent	5 years +	1. All skills of Lab Technician II 2. Experienced in training staff
Lab Technician/Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician/Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Chemist I 2. Trained in majority of department methods

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Lab Technician/Chemist III	BS/BA in Chemistry, Biology, Environmental or related Science	5 years +	1. All skills of Chemist II 2. Experienced in training staff
Analyst I	HS or Equivalent	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analyst II	HS or Equivalent	2-4 years	1. All skills of Analyst I 2. Experienced in machine operation, maintenance and troubleshooting
Analyst III	HS or Equivalent	5 years +	1. All skills of Analyst II 2. Experienced in data review and reporting 3. Experienced in training staff
Analytical Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	6 mos-1 year	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analytical Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Analytical Chemist I 2. Experienced in machine operation, maintenance and troubleshooting
Analytical Chemist III	BS/BA in Chemistry, Biology, or Environmental or related Science	5 years +	1. All skills of Analytical Chemist II 2. Experienced in data review and reporting 3. Experienced in training staff
Data Deliverable Specialist I	HS Diploma, BS/BA or Associates preferred	0-1 years	1. Introductory knowledge of laboratory methods 2.Able to follow direction and Standard Operating Procedures (SOP's) 3. Working knowledge of Adobe Acrobat, Microsoft Word, Excel 4. Good writing and typing skills
Data Deliverable Specialist II	HS Diploma, BS/BA or Associates preferred	2-4 years	1. All skills of Data Deliverable Specialist I 2. General knowledge of laboratory methods 3. Understanding of data review/ data reporting process 4. Experience with and understanding of LIMS and electronic data deliverables

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Data Deliverable Specialist III	HS Diploma, BS/BA or Associates preferred	5 years +	1. All skills of Data Deliverable Specialist II 2. Intermediate/advanced knowledge of laboratory methods 3. Able to perform report review 4. Experience with and understanding of LIMS and electronic data deliverables 5. Able to initiate re-work where necessary
Laboratory Intern	2 Semesters of Chemistry, Biology or Environmental Science	None; Lab work study experience preferred	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures

KEY

* Internal terms only. Full title would have "Environmental Laboratory" and specific department preceding it.

** Substitutions: Equivalent knowledge may be substituted for a degree in some instances.

*** Not meant to be an exhaustive list of skill requirements. For full list of skills consult the "Laboratory Skills" list. Actual Job Duties and Responsibilities can be found within job descriptions for each position.

24 Appendix I – Standard Operating Procedures

WESTBORO SOP #	Title
1728	Waste Management and Disposal
1730	Balance Calibration Check
1733	Thermometer Calibration
1735	Analytical Guidelines for Method Validation
1737	Inorganics Glassware Cleaning and Handling
1738	Water Quality Monitoring
1745	Reagent, Solvent and Standard Control
1747	Datalogger Operation
1948	Separatory Funnel Liquid-Liquid Extraction – EPA 3510C
1953	Organic Extraction Glassware Cleaning & Handling
1954	Soxhlet Extraction – EPA 3540C
1955	Sulfur Cleanup – EPA 3660A
1956	Oil and Waste Dilution – EPA 3580A
1959	Microwave Extraction – EPA 3546
1960	Sulfuric Acid Cleanup – EPA 3665A
1962	Florisil Cleanup
1963	Fractionation Cleanup
1964	Preparation of Samples for Chlorinated Herbicides
2022	Volatile Organic Compounds – EPA 624
2107	Volatile Organic Compounds – EPA 524.2
2108	Volatile Organic Compounds – EPA 8260C
2109	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM – EPA 8270D (modified)
2110	Semivolatile Organics by GC/MS – EPA 625
2111	Semivolatile Organics by GC/MS – EPA 8270D
2112	TCLP/SPLP Extraction - Volatile Organics SW-846 Method 1311/1312
2113	EDB & DBCP in Water by Microextraction & Gas Chromatography – EPA 504.1, 8011
2116	Organochlorine Pesticides by Capillary Column GC – EPA 8081B
2119	Extractable Petroleum Hydrocarbons – MADEP
2120	Volatile Petroleum Hydrocarbons – MADEP
2122	Organochlorine Pesticides & PCBs by Capillary Column GC – EPA 608
2123	Polychlorinated Biphenyls in Oil – EPA 600/4-81-045
2125	TPH-Diesel Range Organics, Maine 4.1.25, EPA 8015C (Modified)
2126	TPH- Gasoline Range Organics, Maine 4.2.17, EPA 8015C (Modified)
2127	CT-ETPH

2128	Herbicides by 8151A
2129	PCBs by Capillary Column Gas Chromatography - EPA 8082A
2131	New Jersey EPH Method
2132	Microwave Assisted Acid Digestion of Aqueous Samples & Extracts – EPA 3015
2133	TCLP Extraction Metals and Semi-Volatile Organics – SW-846 Method 1311
2134	Hot Block Digestion for Aqueous Samples EPA 3005A
2135	SPLP Extraction Inorganics and Semivolatile Organics, EPA 1312
2136	Hot Plate Digestion of Sediments, Sludges and Soils, EPA 3050B
2144	Metals by Inductively Coupled Plasma – EPA 6010C
2145	Mercury in Liquid Waste by Cold-Vapor Atomic Absorption – EPA 7470A
2146	Mercury in Soil or Solid Waste by Cold-Vapor AA – EPA 7471B
2149	Metals by Inductively Coupled Plasma – EPA 200.7
2153	Mercury in Water by Automated Cold-Vapor Atomic Absorption – EPA 245.1
2156	Metals by Inductively Coupled Plasma-Mass Spectrometry – EPA 6020A
2159	Metals by Inductively Coupled Plasma-Mass Spectrometry – EPA 200.8
2161	Fecal Coliform by Membrane Filtration – SM 9222D
2163	Fecal Coliform by Multiple Tube Fermentation – SM 9221E
2191	Heterotrophic Plate Count – SM 9215B
2192	Total Coliform/E.Coli – Presence/Absence (Colilert) – SM 9223B
2193	Total Coliform by Membrane Filtration – SM 9222B
2194	Total Coliform by Multiple Tube Fermentation – SM 9221B
2195	Chlorophyll A – SM 10200H
2196	E. Coli – Membrane Filtration
2197	Chlorophyll A – EPA 446
2198	Air Density Monitoring
2199	Inhibitory Residue Test
2200	Enterococcus – MF
2201	Total Coliform, E.Coli & Enterococcus by Quantification Methods (Quanti Tray)
2202	pH, Liquid Samples
2203	pH, Soil & Waste Samples
2204	Hexavalent Chromium
2205	Biological Oxygen Demand
2206	Ammonia Nitrogen
2207	Total Kjeldahl Nitrogen
2208	Chemical Oxygen Demand
2209	Oil & Grease by n-Hexane Extraction Method & Gravimetry
2210	Cyanide, Total

2211	Phenol, Total
2212	Sulfate, Turbidimetric Method
2213	Alkalinity, Titration Method –SM 2320B
2214	Determination of Inorganic Anions by Ion Chromatography – EPA 300.0
2215	Total Organic Carbon/Dissolved Organic Carbon
2216	Chloride – SM 4500Cl-E, EPA 9251
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen – EPA 353.2, SM 4500NO_3-F
2218	Total Solids (Dried @ 103-105°) and TVS – SM 2540B, SM 2540E
2219	Total Dissolved Solids – SM 2540C
2220	Total Suspended Solids – SM 2540D
2221	Total Sulfide – SM 4500S2-AD, EPA 9030B
2222	MBAS, Anionic Surfactants – SM 5540C
2223	Fluoride, Electrode Method – SM 4500F-BC
2224	Turbidity, Nephelometric Method – EPA 180.1, SM 2130B
2225	Orthophosphate, Colorimetric Single Reagent Method – SM 4500P-E
2226	Total Phosphorous, Colorimetric Combined Reagent Method – SM 4500P-E
2227	Flashpoint – EPA 1010
2228	Reactivity – EPA Chapter 7.3
2229	Total Solids (Dried @ 103-105°) – SM 2540G
2230	Specific Conductance and Salinity
2231	True and Apparent Color, Visual Comparison Method
2232	Acidity, Titration Method
2233	Determination of Formaldehyde by HPLC, EPA 8315A
2234	Sulfite, Iodometric
2235	Ferrous Iron
2236	Residual Chlorine
2237	ORP
2238	Ignitability of Solids EPA 1030
2239	Physiologically Available Cyanide (PAC)
2240	Total Settleable Solids SM 2540 F
2241	Fixed and Volatile Solids in Solid and Semisolid Samples – SM 2540G
2242	Tannin & Lignin
2243	Nitrite - Manual Colorimetric Method
2244	Paint Filter Liquids Test
2245	Odor, Threshold Odor Test
2249	Dissolved Oxygen
2251	Perchlorate by IC/MS/MS
2274	Data Validation Package Generation
3743	Free Cyanide
9177	Total Phenol - SEAL Method

9733	Oil & Grease and TPH in Soil
10807	Percent Organic Matter in Soil
12838	Buchi Concentration
17620	Microwave Digestion for Metals EPA 3015A/3051A
17972	Extractable Organic Halides (EOX)
18236	Chloropicrin and Carbon Tetrachloride by EPA 8011

MANSFIELD SOP #	Title
1753	Glassware Cleaning
1754	Balance Calibration
1755	Pipette Checks
1796	Sample Management - Forensics
1797	Haz Waste
1816	Reagent Solvent Standard Control
2137	ICP-MS EPA 6020A
2138	Mercury Aqueous 7470A
2139	Mercury Soil 7471B
2140	AVS SEM
2141	Hydride Generation
2142	Mercury Aqueous 1631E
2143	Mercury Soil 7474
2148	Metals Soil Digestion 3050
2150	Metals Microwave 3015
2151	Metals Acid Digestion 3020
2152	Seawater Extraction of Metals
2154	TCLP 1311
2155	EPA 8270D
2157	PAH by SIM
2158	EPA 8081B
2160	EPA 8082A Aroclors/Congeners by GC
2162	Pesticides/PCB Aroclors/Congeners by GC/MS SIM
2164	1,4-Dioxane GC/MS SIM
2165	Separatory Funnel Extraction EPA 3510C
2166	Tissue Prep
2167	GPC
2168	Sulfur Cleanup 3660
2169	Sulfuric Acid Cleanup 3665
2170	Silica Gel Cleanup

2171	% Lipids
2172	Microscale Solvent Extraction EPA 3570
2173	Soxhlet Extraction EPA 3540C
2174	Soxhlet Extraction of PUFs
2175	% Total Solids
2182	TOC by Lloyd Kahn
2183	Particle Size Determination
2184	Particulates in Air PM-10
2185	Volatile Solids
2186	TO-15
2187	APH
2188	Air PIANO
2189	Dissolved Gases
2190	Can Cleaning
2246	TPH and SHC
2247	Alkylated PAH
2248	Organic Lead
2252	Fixed Gases
2253	TO-11A
2255	PIANO Volatiles
2256	Ethanol in Oil
2257	Whole Oil Analysis
2259	Density Determination of Oils
2260	Alumina Cleanup
2261	Shaker Table
2263	Gravimetric Determination
2264	Tissue Extraction
2265	Organic Waste Dilution
2267	Client SOP: SGC - Manual Method
2268	Client SOP: DCM Extractable Method
4246	PAHs by SPME
6398	TO-17
6438	Mercury in Sorbent Tubes by CVAA
7900	Mercury 1631E Using Cetac-M-8000 Analyzer
9077	Porewater Generation
9480	EPA-TO-12
9745	Formaldehyde - HPLC
12863	EPA 8270D GC/MS Full Scan TO-13A
13091	HPAH
13392	EPA TO-10A
13406	Particulate Organic Carbon

14500	Lead in Particulate Matter
17452	TOC by EPA 9060A
17456	Moisture, Ash and Organic Matter
18086	Total Suspended Solids (TSS) SM 2540D

CORPORATE SOP #	Title
1559	Sample Receipt and Login
1560	Sample Custody and Tracking
1561	Bottle Order Preparation
1562	Computer System Backup/Control
1563	Computer and Network Security
1564	Software Validation and Control
1565	Training Program
1566	Report Generation and Approval
1567	Organics Data Deliverable Package Review
1722	Customer Inquiry and Complaint Procedures
1723	Customer Service
1724	Quote/Contract Procedure
1725	Project Communication Form Generation
1726	Purchasing Procedure
1727	Accounts Payable Invoice Processing
1729	Document Control
1731	Manual Integration and Compound Rejection
1732	DL LOD LOQ Generation
1734	Control Limit Generation
1736	Corrective and Preventative Actions
1739	Demonstration of Capability (DOC) Generation
1740	Internal Audit Procedure
1741	Data Review – Organics
1742	Calculating Measurement Uncertainty
1743	Annual Management Review
1744	Sample Compositing Procedure
1746	Nonconformance Planning/Procedures
1747	Temperature Datalogger Operation
2274	Data Validation Package
17553	Lab Supply Transfer Procedure

Appendix 2: Laboratory Standard Operating Procedures

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, 2006.

Method 5035A, Closed System Purge &Trap and Extraction for Volatile Organics in Soil and Waste Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, Draft, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December, 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May, 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The following compounds may be determined by this method:

8260C LIST OF ANALYTES		
Dichlorodifluoromethane	Carbon tetrachloride	Isopropylbenzene
Chloromethane	1,2-Dichloroethane	1,4-Dichloro-2-butane
Vinyl chloride	Benzene	1,1,2,2-Tetrachloroethane
Chloroethane	Trichloroethene	Trans-1,4-dichloro-2-butene
Bromomethane	1,2-Dichloropropane	1,2,3-Trichloropropane
Trichlorofluoromethane	Bromodichloromethane	n-Propylbenzene
Ethyl ether	Dibromomethane	Bromobenzene
Acetone	4-Methyl-2-pentanone	2-Chlorotoluene
1,1-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
Carbon disulfide	Toluene	4-Chlorotoluene
Methylene chloride	Trans-1,3-dichloropropene	Tert-butylbenzene
Acrylonitrile	Ethyl-methacrylate	1,2,4-Trimethylbenzene
Methyl-tert-butyl ether	1,1,2-Trichloroethane	Sec-butylbenzene
Trans-1,2-dichloroethene	2-Hexanone	p-Isopropyltoluene
1,1-Dichloroethane	1,3-Dichloropropane	1,3-Dichlorobenzene
Vinyl acetate	Tetrachloroethene	1,4-Dichlorobenzene
2-Butanone	Chlorodibromomethane	n-Butylbenzene
2,2-Dichloropropane	1,2-Dibromoethane	1,2-Dichlorobenzene
Cis-1,2-dichloroethene	Chlorobenzene	1,2-Dibromo-3-chloropropane
Chloroform	1,1,1,2-Tetrachloroethane	1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl benzene	Hexachlorobutadiene
Tetrahydrofuran	p/m Xylene	Naphthalene
1,1,1-Trichloroethane	o Xylene	1,2,3-Trichlorobenzene

8260C LIST OF ANALYTES (continued)		
1,1-Dichloropropene	Styrene	Bromoform
Acrolein	2-Chloroethylvinyl ether	Ethanol
Cyclohexanone	Ethyl acetate	1,3,5-Trichlorobenzene
Iodomethane	Methyl methacrylate	Tert-amyl methyl ether
Di-isopropyl ether	n-Butanol	1,4-Dioxane
Ethyl Tert-Butyl Ether	Pentachloroethane	Isopropyl Alcohol (IPA)

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column 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 (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 10.3 and 9.1

demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally all affected samples are rerun for confirmation.. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab, for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below, but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures. The Lab will analyze the high level sample first, followed by the low level sample if the results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol, and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.1.2.2.2 and 11.1.2.2.3)

6.1.2.2 Low Level Soil Samples

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Collect sample in a standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedence, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples requiring aromatic analysis must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples and High Level Soils): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (Varian Archon/8100, Tekmar Solatek, EST Centurion) coupled to the desorber (concentrator) (Tekmar Velocity or EST Encon).

7.1.1 Purge gas = Helium, analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the helium purge gas is introduced at the bottom of the water column as finely divided bubbles

7.1.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260°C. The trap is not heated above manufacturer’s specifications

7.2. Purge and Trap System (For Low Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) coupled to the desorber (concentrator) (Varian Archon/8100, Tekmar Solatek, EST Centurion with EST Encon, Tekmar Velocity, or equivalents).

7.2.1. Purge gas = Helium, analytical grade (99.999%).

7.2.2. The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the helium purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3. The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.2.4. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, or equivalent.
7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, or equivalent

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Scanning from 35 to 300 amu every 2 seconds 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 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition, and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

Thruput Target 4.12 software or Enviroquant E.02.02 (or equivalent) is used for data processing, and allows searching of any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / Enviroquant data system.

7.4 Wiretrol or Microsyringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.1g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf Pipets

8. Reagents and Standards

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

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards:

Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in the beginning of this SOP. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 initial calibration standard. This standard is made from a second source than the Initial Calibration Standards.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Blank samples must be matrix specific, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed; TCLP samples need a TCLP blank.

Analyze a matrix-specific blank each day prior to sample analysis to demonstrate that interferences from the analytical system are under control. The blank must contain the internal standards and surrogates.

Analyze the reagent water blank from the same source of water used for preparing the standards, QC samples and making sample dilutions. The method blank must not contain any target analytes at or above the compound reporting limits.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

A LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.4.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run anytime after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium, 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass scan range:	35 – 260 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	1 minute
Desorb preheat:	245°C
Desorb temp:	255°C
Desorb time:	1 minute
Bake temp:	270°C
Bake time:	10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level of the either at or below the reporting limit. The calibration is performed using instrument conditions listed in Section 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards, and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the ThruPut Target / Enviroquant software.

10.2.1.1 **Low Level/High Level Soil Curve on Archon or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto Archon Autosampler.

10.2.1.2 **Aqueous/High Level Soil Curve on Solatek or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 40mL VOA vial to the top. Load the vial onto the Autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP Enviroquant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the Target / Enviroquant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the Target / Enviroquant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 **Initial Calibration Criteria:** The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed.

10.2.3.1 **Relative Standard Deviation Criteria:** If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to

quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if **r** $\geq$ 0.990, weighted linear with a weighting factor of 1/SD2 and r > 0.990, or quadratic fit if $\mathbf{r^2} \geq 0.995$. A minimum of six points is required and the low point of the calibration must be re-quantitated and recover within 70-130% to be deemed acceptable. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.2 **Minimum Response Factors:** Table 1 lists the suggested minimum response factors for the most common analytes. Each calibration level must be evaluated against the specified criteria. Analytes that fall below the criteria, but are greater than or equal to 0.05, are narrated for inclusion on the final report. There are certain very poor purgers (1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds) that should meet a 0.001 response factor. If an analyte falls below 0.05 (or 0.001 for 1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds), then corrective action must be taken to resolve the problem before analysis can proceed.

10.2.4 **Evaluation of Retention Times:** The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 **Initial Calibration Verification:** After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes except for “difficult” analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 Analysis of Samples

Retrieve sample VOA vials from the sample bank refrigerator just prior to loading onto the purge and trap system. High level soil samples must be shaken for 1 – 2 minutes to extract the volatile components into the methanol. Let sample settle prior to taking methanol aliquot. Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the Archon or Centurion unit.

10.3.1.1 **Low level soil samples:** (Archon or Centurion)
Take the low level VOA vial and place directly into the rack of the Archon sampling unit. Surrogate and internal standards are added automatically by the Archon prior to sample purging.

10.3.1.2 **Aqueous samples:** (Solatek or Centurion)

Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The auto-sampler will then sample 10mL from the VOA vial.

10.3.1.3 High level soil samples: (Archon/Solatek/Centurion)

Shake for 2 minutes, ensuring the methanol has completely penetrated the soil in the vial.

10.3.1.3.1 Through liquid path
Load a maximum of 430µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.1.3.2 Through soil path
Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.2 Qualitative Analysis:

10.3.2.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.2.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The Target / Enviroquant data system is configured to make this check.

10.3.2.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.2.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.2.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

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

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

10.3.2.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as “tentative”, and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

1) 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.
2) 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%.)
3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3 Quantitative Analysis:

10.3.3.1 Quantitation of a target compound detected in a sample is performed automatically by the Target / Enviroquant data processing software, using the formulae found in Alpha’s Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve.

If non-target compounds are to be reported, the quantitation is performed automatically by the Target / Enviroquant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 The continuing calibration standard must also be evaluated for the suggested minimum response factor criteria, as specified in section 10.2.3.2

10.4.6 Internal Standard Retention Time:

The retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.7 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.1.2 Data Calculations

11.1.2.1 Results of Aqueous Sample Analysis:

$$\text{concentration (ug/L)} = \frac{(Conc.)\,(Vp)\,(DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.1.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.1.2.2.1 Low-Level Samples:

$$\text{concentration (ug/Kg)} = \frac{(Conc.)\,(Vp)\,(DF)}{(W)\,(\%S)}$$

11.1.2.2.2 High-Level Samples:

$$\text{concentration (ug/Kg)} = \frac{(Conc.)\,(Vp)\,(5000)\,(DF)}{(W)\,(Ve)\,(\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.1.2.2.3 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\%\ moisture = \frac{g\ of\ sample - g\ of\ dry\ sample}{g\ of\ sample} \times 100$$

$$V_t = \frac{[mL\ of\ solvent + (\%moisture \times g\ of\ sample)]}{100} \times 1000mL/mL$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$Corrected\ concentration\ (mg/Kg) = \frac{(Conc.)\ (V_t + methanol\ vol.)\ (Vp)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: 8260 REPORTING LIMITS

TABLE 2: 8260 QC ACCEPTANCE CRITERIA

TABLE 3: BFB TUNING CRITERIA

TABLE 4: STANDARD SOLUTIONS

TABLE 5: 8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 6: 8260C Quantitation Ions

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
Acetone (3,4,5)	0.100	5.0	10	250
Acrolein (5)		5.0	25	1250
Acrylonitrile (3,4)		5.0	5	200
Benzene (3,4,5)	0.500	0.5	1	50
Bromobenzene (3,4)		2.5	5	250
Bromochloromethane (3,4,5)		2.5	5	250
Bromodichloromethane (3,4,5)	0.200	0.5	1	50
Bromoform (3,4,5)	0.100	2.0	4	200
Bromomethane (3,4,5)	0.100	1.0	2	100
2-Butanone (3,4,5)	0.100	5.0	10	500
n-Butyl benzene (3,4)		0.5	1	50
sec-Butyl benzene (3,4)		0.5	1	50
tert-Butyl benzene (3,4)		2.5	5	250
Carbon disulfide (3,4,5)	0.100	5.0	10	500
Carbon tetrachloride (3,4,5)	0.100	0.5	1	50
Chlorobenzene (3,4,5)		0.5	1	50
Chloroethane (3,4,5)	0.100	1.0	2	100
2-Chloroethylvinyl ether (3)		10.0	20	1000
Chloroform (3,4,5)	0.200	0.75	1.5	75
Chloromethane (3,4,5)	0.100	2.5	5	250
o-Chlorotoluene (3,4)		2.5	5	250
Cyclohexane (5)	0.100	10	20	1000
Cyclohexanone		10	20	1000
p-Chlorotoluene (3,4)		2.5	5	250
Dibromochloromethane (3,4,5)	0.100	0.5	1	50
1,2-Dibromo-3-chloropropane (3,4,5)	0.050	2.5	5	250
1,2-Dibromoethane (3,4,5)	0.100	2.0	5	250
Dibromomethane (3,4)		5.0	10	500
1,2-Dichlorobenzene (3,4,5)	0.400	2.5	5	250
1,3-Dichlorobenzene (3,4,5)	0.600	2.5	5	250
1,4-Dichlorobenzene (3,4,5)	0.500	2.5	5	250
1,4-Dichlorobutane (3,4)		5.0	10	500
trans-1,4-Dichloro-2-butene (3,4)		2.5	5	250
Dichlorodifluoromethane (3,4,5)		5.0	10	500
1,1-Dichloroethane (3,4,5)	0.200	0.75	1.5	75
1,2-Dichloroethane (3,4,5)	0.100	0.5	1	50
1,1-Dichloroethene (3,4,5)	0.100	0. 5	1	50
cis-1,2-Dichloroethene (3,4,5)	0.100	0.5	1	50
trans-1,2-Dichloroethene (3,4,5)	0.100	0.75	1.5	75

Table 1 (continued)
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
1,2-Dichloropropane (3,4,5)	0.100	1.75	3.5	175
1,3-Dichloropropane (3,4)		2.5	5	250
2,2-Dichloropropane (3,4)		2.5	5	250
1,1-Dichloropropene (3,4)		2.5	2.5	250
cis-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
p-Diethylbenzene (4)		2.0	4	200
Diisopropyl Ether (6)		2.0	4	200
1,4-Dioxane (5) (non-SIM)		250	100	5000
trans-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
Ethanol (7)		N/A	1000	50000
Ethyl acetate		10.0	20	1000
Ethylbenzene (3,4,5)	0.100	0.5	1	50
Ethyl ether (3,4)		2.5	5	250
4-Ethyltoluene (4)		2.0	4	200
Ethyl methacrylate (3,4)		5.0	10	500
Ethyl-Tert-Butyl-Ether (6)		2.0	4	200
Freon-113 (5)		10.0	20	1000
Hexachlorobutadiene (3,4)		0.5	5	250
2-Hexanone (3,4,5)	0.100	5.0	10	500
Iodomethane		5.0		
Isopropyl Alcohol (IPA)		25		
Isopropylbenzene (3,4,5)	0.100	0.5	1	50
p-Isopropyltoluene (3,4)		0.5	1	50
Methyl Acetate (5)	0.100	20	20	1000
Methylene chloride (3,4,5)	0.100	3.0	10	500
Methyl Cyclohexane (5)	0.100	20	4	200
Methyl Methacrylate		1.0		
4-Methyl-2-pentanone (3,4,5)	0.100	5.0	10	500
Methyl-tert-butyl-ether (3,4,5)	0.100	1.0	2	100
Naphthalene (3,4)		2.5	5	250
n-Butanol (5)		100	200	10000
n-Propylbenzene (3,4)		0.5	1	50
Pentachloroethane		2.0	N/A	N/A
Styrene (3,4,5)	0.300	1.0	2	100
Tert-Butyl Alcohol (5)		30	100	5000
Tertiary-Amyl Methyl Ether (6)		2.0	4	200
1,1,1,2-Tetrachloroethane (3,4)		0.5	1	50
1,2,4,5-Tetramethylbenzene (4)		2.0	4	200

Analyte	**Recommended Minimum Response Factor**	**RDL (µg/L)**	**RDL(µg/KG)(1)**	**RDL(µg/KG)(2)**
1,1,2,2-Tetrachloroethane (3,4,5)	0.300	0.5	1	50
Tetrachloroethene (3,4,5)	0.200	0.5	1	50
Tetrahydrofuran (3)		10.0	20	1000
Toluene (3,4,5)	0.400	0.75	1	75
1,2,3-Trichlorobenzene (3,4,5)		2.5	5	250
1,2,4-Trichlorobenzene (3,4,5)	0.200	2.5	5	250
1,3,5-Trichlorobenzene (6)		2.0	5	250
1,1,1-Trichloroethane (3,4,5)	0.100	0.5	1	50
1,1,2-Trichloroethane (3,4,5)	0.100	0.75	1.5	75
Trichloroethene (3,4,5)	0.200	0.5	1	50
Trichlorofluoromethane (3,4,5)	0.100	2.5	5	250
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	5	250
1,3,5-Trimethylbenzene (3,4)		2.5	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.100	1.0	2	100
m/p-Xylenes (3,4,5)	0.100	1.0	2	100
o-Xylene (3,4,5)	0.300	1.0	2	100
1,4-Dioxane (5) SIM		3.0		
1,1,2,2-Tetrachloroethane SIM		0.1		

(1) Detection Limits are for Low-level Aqueous preserved samples.
(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.

Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for New York and New Jersey reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Acetone	2000	0.5	2	10	20	30	50	100	200
Acrolein	2000	0.5	2	10	20	30	50	100	200
Acrylonitrile	2000	0.5	2	10	20	30	50	100	200
Benzene	2000	0.5	2	10	20	30	50	100	200
Bromobenzene	2000	0.5	2	10	20	30	50	100	200
Bromochloromethane	2000	0.5	2	10	20	30	50	100	200
Bromodichloromethane	2000	0.5	2	10	20	30	50	100	200
Bromoform	2000	0.5	2	10	20	30	50	100	200
Bromomethane	2000	0.5	2	10	20	30	50	100	200
2-Butanone	2000	0.5	2	10	20	30	50	100	200
n-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
sec-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
tert-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
Carbon disulfide	2000	0.5	2	10	20	30	50	100	200
Carbon tetrachloride	2000	0.5	2	10	20	30	50	100	200
Chlorobenzene	2000	0.5	2	10	20	30	50	100	200
Chloroethane	2000	0.5	2	10	20	30	50	100	200
2-Chloroethylvinyl Ether	2000	0.5	2	10	20	30	50	100	200
Chloroform	2000	0.5	2	10	20	30	50	100	200
Chloromethane	2000	0.5	2	10	20	30	50	100	200
o-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
p-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Cyclohexanone	2000	0.5	2	10	20	30	50	100	200
Dibromochloromethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromo-3-chloropropane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromoethane	2000	0.5	2	10	20	30	50	100	200
Dibromomethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobutane	2000	0.5	2	10	20	30	50	100	200
trans-1,4-Dichloro-2-butene	2000	0.5	2	10	20	30	50	100	200
Dichlorodifluoromethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
cis-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
trans-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,3-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
2,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloropropene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
cis-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
trans-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
p-Diethylbenzene	2000	0.5	2	10	20	30	50	100	200
Diisopropyl Ether	2000	0.5	2	10	20	30	50	100	200
1,4-Dioxane (non-SIM)	10000	100	400	1000	2000	3000	4000	5000	6000
Ethanol	10000	100	200	300	500	1000	2500	5000	N/A
Ethyl Acetate	2000	0.5	2	10	20	30	50	100	200
Ethylbenzene	2000	0.5	2	10	20	30	50	100	200
Ethyl ether	2000	0.5	2	10	20	30	50	100	200
Ethyl methacrylate	2000	0.5	2	10	20	30	50	100	200
Ethyl Tert-Butyl Ether	2000	0.5	2	10	20	30	50	100	200
4-Ethyltoluene	2000	0.5	2	10	20	30	50	100	200
Freon-113	2000	0.5	2	10	20	30	50	100	200
Halothane	2000	0.5	2	10	20	30	50	100	200
Hexachlorobutadiene	2000	0.5	2	10	20	30	50	100	200
2-Hexanone	2000	0.5	2	10	20	30	50	100	200
Iodomethane	2000	0.5	2	10	20	30	50	100	200
Isopropyl Alcohol (IPA)	10000	2.5	10	50	100	150	250	500	1000
Isopropylbenzene	2000	0.5	2	10	20	30	50	100	200
p-Isopropyltoluene	2000	0.5	2	10	20	30	50	100	200
Methyl Acetate	2000	0.5	2	10	20	30	50	100	200
Methylene Chloride	2000	0.5	2	10	20	30	50	100	200
Methyl Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Methyl Methacrylate	2000	0.5	2	10	20	30	50	100	200
4-Methyl-2-pentanone	2000	0.5	2	10	20	30	50	100	200
Methyl-tert-butyl-ether	2000	0.5	2	10	20	30	50	100	200
Naphthalene	2000	0.5	2	10	20	30	50	100	200
n-Butanol	5000	2.5	10	50	100	150	250	500	N/A
n-Propylbenzene	2000	0.5	2	10	20	30	50	100	200
Pentachloroethane	1000	0.5	2	10	20	30	50	100	200
Styrene	4000	1	4	20	40	60	100	200	400
Tert-Butyl alcohol	10000	2.5	10	50	100	150	250	500	1000
Tertiary-Amyl Methyl Ether	2000	0.5	2	10	20	30	50	100	200
1,1,1,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
Tetrachloroethene	2000	0.5	2	10	20	30	50	100	200
Tetrahydrofuran	2000	0.5	2	10	20	30	50	100	200
1,2,4,5-Tetramethylbenzene	2000	0.5	2	10	20	30	50	100	200
Toluene	2000	0.5	2	10	20	30	50	100	200
1,2,3-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
1,2,4-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,1,1-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
Trichloroethene	2000	0.5	2	10	20	30	50	100	200
Trichlorofluoromethane	2000	0.5	2	10	20	30	50	100	200
1,2,3-Trichloropropane	2000	0.5	2	10	20	30	50	100	200
1,2,4-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
Vinyl acetate	2000	0.5	2	10	20	30	50	100	200
Vinyl chloride	2000	0.5	2	10	20	30	50	100	200
m/p-Xylenes	4000	1	4	20	40	60	100	200	400
o-Xylene	4000	1	4	20	40	60	100	200	400
1,4-Dioxane (SIM)	100	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane (SIM)		0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0

Target Compounds	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Internal Standards									
Fluorobenzene	2500	10	10	10	10	10	10	10	10
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10
Surrogates									
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

TABLE 5

8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile		p-isopropyltoluene
Methyl Tert Butyl Ether		1,3-dichlorobenzene
Halothane		1,4-dichlorobenzene
Trans-1,2-dichloroethene		n-butylbenzene
Diisopropyl Ether		p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		1,3,5-Trichlorobenzene
1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		
1,1-dichloropropene		
Carbon Tetrachloride		
Tertiary-Amyl Methyl Ether		
1,2-dichloroethane-d4 (surr)		
1,2-dichloroethane		
Benzene		
Trichloroethene		
Methyl Cyclohexane		
1,2-dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
Cis-1,3-dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl Alcohol (IPA)		

TABLE 6
8260C Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-tetramethylbenzene	119
Trichloroethene	95	1,2-dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-trichlorobenzene	180
1,2-dichloropropane	63	1,2,4-trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

TABLE 6
8260C Quantitation Ions (continued)

Analyte	Quantiation Ion	Analyte	Quantiation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
naphthalene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: **EPA 8270 D**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists “difficult” compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953.

4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2ºC.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 Column: Rxi-5Sil MS30m x 0.32mm ID, 0.25µm film thickness or column of similar configuration.

7.1.3 Mass Spectrometer, Hewlett Packard 5973 (or equivalent): Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 Data System: A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *Target / NT Revision 4.12* is used for data reduction.

7.2 Syringe: 10 µL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer.

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Vendor	Standard	Catalog No.	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/ml
	605 Benzidines Mix	31030	2000ug/ml
	Benzoic Acid Mix	31879	2000ug/ml
	Acid Surrogate Mix	31025	2000ug/ml
	Acid Surrogate Mix	31087	10000ug/ml
	BN Surrogate Mix	31024	1000ug/ml
	B/N Surrogate Mix	31086	5000ug/ml
	Custom SV Standard	562843	2000ug/ml
	Custom ABN Addition Standard	567302	2000ug/ml
	Benzaldehyde Standard	33017	2000ug/ml
	Alpha-Terpineol Standard	33912	2000ug/ml
	8270 Benzidines Mix#2	31852	2000ug/ml
Absolute	Atrazine	70023	1000ug/ml
AccuStandard	Parathion	M-622-19	1000ug/ml
	2,6-Dichlorophenol	M-8040-08	1000ug/ml
SPEX	n-Decane	S-1115	1000ug/ml
	n-Octadecane	S-2850	1000ug/ml
	2,6-Dichlorophenol	S-1415	1000ug/ml
	a-Terpineol	S-3356-AC	1000ug/ml
	Diesel Range Organics Mix	DRO-1000	1000ug/ml
	Custom SVOA	SVO-ALAMA-7-4	2000ug/ml
Ultra	Atrazine	EPA-1176A	1000ug/ml
	Parathion	SP-140-1	100ug/ml

8.1.1 ABN Mega Mix Standard, 200µg/mL

Use 5mL of each of the following:
605 Benzidines Mix
Benzoic Acid Mix
Acid Surrogate Mix

and use 10mL of each of the following:
8270 Mega Mix

and use 2mL of each of the following:
B/N Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Standard, 200ug/mL

Use 5mL of each of the following:

Custom SV Standard
Custom ABN Addition Standard
Benzaldehyde Standard

and use 10mL of each of the following:
a-Terpineol Standard
2,6-Dichlorophenol

Bring up to 50mL volume with DCM.

8.1.3 ADP Standard, 200ug/ml

Use 5ml of:
8270 Benzidines Mix#2

and use 10mL of each of the following:
Parathion
Atrazine
n-Decane
n-Octadecane

Bring up to 50mL volume with DCM.

8.1.4 Calibration Stock Standards Preparation

A minimum of 5 calibration standards for each analyte are prepared.

Level	***Concentration (ug/mL)***
L1	1
L2	2
L3	3
L4	5
L5	10
L6	20
L7	50
L8	100
L9	150
L10	200

8.2 Internal Standard Solution

This is a premixed, certified solution from Supelco, 2000ng/mL in DCM, catalog #4-8902. Each 500µL of standards, blank and sample extracts are spiked with 10µL of Internal Standard Solution, resulting in a concentration of 40ng/µL.
The internal standards are: 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12}.

8.3 GC/MS Tuning Standard

A methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP) is used for checking the tune. The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

This working standard is prepared from a stock solution, purchased from Ultra Scientific, Catalog# GCM-150.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL DCM.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml each of Base-Neutrals Surrogate Mix #31086 and Acid Surrogate Mix #31087. Bring up to volume with Acetone. The final concentration is 50ug/ml for the Acid surrogates and 25ug/ml for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

8.5.1 Spike Solution Preparation

ABN SPK1:

In a 200ml volumetric flask add 8ml of 8270 Mega Mix #31850, 4ml of Benzoic Acid Mix#31879, Custom SV Standard#562843, Benzaldehyde Standard#33017, Custom SVOA#SVOA-ALAMA-7-4; Bring up to volume with Acetone. The final concentration is 40ug/ml.

ABN SPK2:

In a 200ml volumetric flask add 8ml of Atrazine#EPA-1176A and Parathion#M-622-19 and 4ml of 8270 Benzidine Mix#2 #31852; Bring up to volume with Acetone. The final concentration is 40ug/ml.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extracion could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with “B” flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based on in-house control limits. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on in-house control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
  - (1) ADP 50 ppm
  - (2) AP9 50 ppm
  - (3) ABN 50 ppm
- Method Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min

Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Target data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, 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.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be ≤ 20% for the mean response factor to be used for sample quantitation.

An alternate calculation fits may be performed provided that the minimum correlation coefficient ≥ 0.99 is met.

When linear regression model is used a minimum quantitation check of the lowest calibration point is performed. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

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10.3 Equipment Operation and Sample Processing

GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500µL of sample extract to another vial, and add 10µL of the internal standard solution (Section 8.2).

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq$ 20%, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Alpha SOP/1732 DL/LOD/LOQ Generation

Alpha SOP/1739 IDC/DOC Generation

Alpha SOP/1729 Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria
Table 9: Difficult analytes

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	**Nitrobenzene-d_5**	**23-120%**	**23-120%**
BN-8270D	**2-Fluorobiphenyl**	**15-120%**	**30-120%**
BN-8270D	**p-Terphenyl-d_{14}**	**41-149%**	**18-120%**
Acid-8270D	**Phenol-d_6**	**10-120%**	**10-120%**
Acid-8270D	**2-Fluorophenol**	**21-120%**	**25-120%**
Acid-8270D	**2,4,6-Tribromophenol**	**10-120%**	**10-136%**

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A

ACCEPTABLE AQUEOUS QC LIMITS

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene			70-130	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20
1,2-Dichlorobenzene	40-140	30	70-130	20
1,3-Dichlorobenzene	40-140	30	70-130	20
1,3-Dinitrobenzene	15-130	30		
1,4-Dichlorobenzene	36-97	30	70-130	20
1-Methylnaphthalene	41-103	30		
2,3,4,6-Tetrachlorophenol			70-130	20
2,4,5-Trichlorophenol	30-130	30	70-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20
2,4-Dichlorophenol	30-130	30	70-130	20
2,4-Dimethylphenol	30-130	30	70-130	20
2,4-Dinitrophenol	20-130	30	20-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20
2,6-Dinitrotoluene	40-140	30	70-130	20
2-Chloronaphthalene	40-140	30	70-130	20
2-Chlorophenol	27-123	30	70-130	20
2-Methylnaphthalene	40-140	30	70-130	20
2-Methylphenol	30-130	30	70-130	20
2-Nitroaniline	52-143	30	70-130	20
2-Nitrophenol	30-130	30	70-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20
3,3'-Dimethylbenzidine			20-160	20
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20
3-Nitroaniline	25-145	30	70-130	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20
4-Chloroaniline	40-140	30	20-160	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20
4-Nitroaniline	51-143	30	70-130	20
4-Nitrophenol	10-80	30	20-160	20
Acenaphthene	37-111	30	70-130	20
Acenaphthylene	45-123	30	70-130	20
Acetophenone	39-129	30	70-130	20
Aniline	40-140	30	20-160	20
Anthracene	40-140	30	70-130	20
Atrazine			70-130	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Azobenzene	40-140	30	70-130	20
Benzaldehyde			20-160	20
Benzidine	10-75	30	20-160	20
Benzo(a)anthracene	40-140	30	70-130	20
Benzo(a)pyrene	40-140	30	70-130	20
Benzo(b)fluoranthene	40-140	30	70-130	20
Benzo(ghi)perylene	40-140	30	70-130	20
Benzo(k)fluoranthene	40-140	30	70-130	20
Benzoic Acid	10-164	30	20-160	20
Benzyl Alcohol	26-116	30	20-160	20
Biphenyl	40-140	30	70-130	20
Bis(2-chloroethoxy)methane	40-140	30	70-130	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20
Butyl benzyl phthalate	40-140	30	70-130	20
Caprolactam			20-160	20
Carbazole	55-144	30	70-130	20
Chrysene	40-140	30	70-130	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20
Dibenzofuran	40-140	30	70-130	20
Diethyl phthalate	40-140	30	70-130	20
Dimethyl phthalate	40-140	30	70-130	20
Di-n-butylphthalate	40-140	30	70-130	20
Di-n-octylphthalate	40-140	30	70-130	20
Fluoranthene	40-140	30	70-130	20
Fluorene	40-140	30	70-130	20
Hexachlorobenzene	40-140	30	70-130	20
Hexachlorobutadiene	40-140	30	70-130	20
Hexachlorocyclopentadiene	40-140	30	20-160	20
Hexachloroethane	40-140	30	20-160	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20
Isophorone	40-140	30	70-130	20
Naphthalene	40-140	30	70-130	20
Nitrobenzene	40-140	30	70-130	20
NitrosoDiPhenylAmine(NDPA)/Diphenylamine (DPA)	40-140	30	70-130	20
n-Nitrosodimethylamine	22-74	30	20-160	20
n-Nitrosodi-n-propylamine	29-132	30	70-130	20
P-Chloro-M-Cresol	23-97	30	70-130	20
Pentachlorophenol	9-103	30	20-160	20
Phenanthrene	40-140	30	70-130	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Phenol	12-110	30	20-160	20
Pyrene	26-127	30	70-130	20
Pyridine	10-66	30		
2-Fluorophenol	*21-120*		*15-110*	
Phenol-d6	*10-120*		*15-110*	
Nitrobenzene-d5	*23-120*		*30-130*	
2-Fluorobiphenyl	*15-120*		*30-130*	
2,4,6-Tribromophenol	*10-120*		*15-110*	
4-Terphenyl-d14	*41-149*		*30-130*	

TABLE 3B

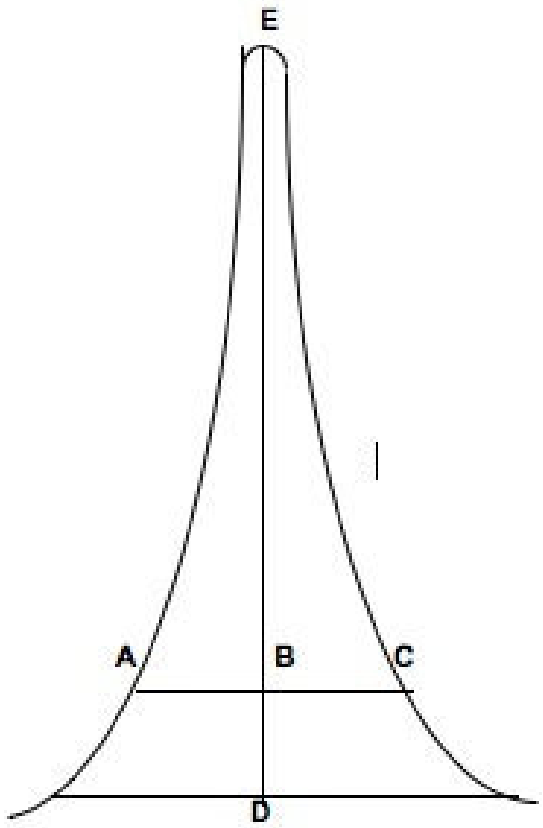
ACCEPTABLE SOIL QC LIMITS

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30
1,2-Dichlorobenzene	40-140	50	70-130	30
1,3-Dichlorobenzene	40-140	50	70-130	30
1,3-Dinitrobenzene	40-140	50		
1,4-Dichlorobenzene	28-104	50	70-130	30
1-Methylnaphthalene	26-130	50		
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30
2,4,5-Trichlorophenol	30-130	50	70-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30
2,4-Dichlorophenol	30-130	50	70-130	30
2,4-Dimethylphenol	30-130	50	70-130	30
2,4-Dinitrophenol	4-130	50	20-160	30
2,4-Dinitrotoluene	28-89	50	70-130	30
2,6-Dinitrotoluene	40-140	50	70-130	30
2-Chloroaniline	30-130	50		
2-Chloronaphthalene	40-140	50	70-130	30
2-Chlorophenol	25-102	50	70-130	30
2-Methylnaphthalene	40-140	50	70-130	30
2-Methylphenol	30-130.	50	70-130	30
2-Nitroaniline	47-134	50	70-130	30
2-Nitrophenol	30-130	50	70-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30
3,3'-Dimethylbenzidine	15-115	50		
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30
3-Nitroaniline	26-129	50	70-130	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30
4-Chloroaniline	40-140	50	20-160	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30
4-Nitroaniline	41-125	50	70-130	30
4-Nitrophenol	11-114	50	20-160	30
Acenaphthene	31-137	50	70-130	30
Acenaphthylene	40-140	50	70-130	30
Acetophenone	14-144	50	70-130	30
Aniline	40-140	50	20-160	30
Anthracene	40-140	50	70-130	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Atrazine	40-140	50	70-130	30
Azobenzene	40-140	50	70-130	30
Benzaldehyde	40-140	50	20-160	30
Benzidine	10-66	50	20-160	30
Benzo(a)anthracene	40-140	50	70-130	30
Benzo(a)pyrene	40-140	50	70-130	30
Benzo(b)fluoranthene	40-140	50	70-130	30
Benzo(e)Pyrene	40-140	50		
Benzo(ghi)perylene	40-140	50	70-130	30
Benzo(k)fluoranthene	40-140	50	70-130	30
Benzoic Acid	10-110	50	20-160	30
Benzyl Alcohol	40-140	50	20-160	30
Biphenyl	54-104	50	70-130	30
Bis(2-chloroethoxy)methane	40-117	50	70-130	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30
Butyl benzyl phthalate	40-140	50	70-130	30
Caprolactam	15-130	50	20-160	30
Carbazole	54-128	50	70-130	30
Chrysene	40-140	50	70-130	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30
Dibenzofuran	40-140	50	70-130	30
Diethyl phthalate	40-140	50	70-130	30
Dimethyl phthalate	40-140	50	70-130	30
Di-n-butylphthalate	40-140	50	70-130	30
Di-n-octylphthalate	40-140	50	70-130	30
Diphenamid	40-140	50		
Fluoranthene	40-140	50	70-130	30
Fluorene	40-140	50	70-130	30
Hexachlorobenzene	40-140	50	70-130	30
Hexachlorobutadiene	40-140	50	70-130	30
Hexachlorocyclopentadiene	40-140	50	20-160	30
Hexachloroethane	40-140	50	20-160	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30
Isophorone	40-140	50	70-130	30
Naphthalene	40-140	50	70-130	30
Nitrobenzene	40-140	50	70-130	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30
n-Nitrosodimethylamine	22-100	50	20-160	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
n-Nitrosodi-n-propylamine	32-121	50	70-130	30
Parathion, ethyl	40-140	50	20-160	30
P-Chloro-M-Cresol	26-103	50	70-130	30
Pentachloronitrobenzene	42-153	50		
Pentachlorophenol	17-109	50	20-160	30
Phenanthrene	40-140	50	70-130	30
Phenol	26-90	50	20-160	30
Pyrene	35-142	50	70-130	30
Pyridine	10-93	50	20-160	30
Thionazin	40-140	50		
2-Fluorophenol	*25-120*		*30-130*	
Phenol-d6	*10-120*		*30-130*	
Nitrobenzene-d5	*23-120*		*30-130*	
2-Fluorobiphenyl	*30-120*		*30-130*	
2,4,6-Tribromophenol	*10-136*		*30-130*	
4-Terphenyl-d14	*18-120*		*30-130*	

TABLE 4 – Tailing Factor Calculation



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	179	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	122	105, 77
Benzyl alcohol	108	79, 77
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	127	129, 65, 92
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
3,3'-Dichlorobenzidine	252	254, 126
2,4-Dichlorophenol	162	164, 98
Diethyl phthalate	149	177, 15

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	212	211, 213
Dimethyl phthalate	163	194, 164
2,4-Dimethylphenol	122	107, 121
Di-n-butyl phthalate	149	150, 104
Di-n-octyl phthalate	149	167, 43
4,6-Dinitro-2-methylphenol	198	51, 105
1,3-Dinitrobenzene	168	76, 50, 75, 92, 122
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
Diphenamide	167	72, 165
Ethyl parathion	109	97, 291
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95, 138
1-Methylnaphthalene	115	141, 142
2-Methylnaphthalene	142	141
2-Methylphenol	107	108, 77, 79, 90
3/4-Methylphenol	107	108, 77, 79, 90
Naphthalene	128	129, 127
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	108, 92
4-Nitroaniline	138	65, 108, 92, 80, 39
Nitrobenzene	77	123, 65
2-Nitrophenol	139	109, 65
4-Nitrophenol	139	109, 65
n-Nitrosodimethylamine	42	74, 44
n-Nitrosodi-n-butylamine	84	57, 41, 116, 158
n-Nitrosodi-n-propylamine	70	42, 101, 130
n-Nitrosodiphenylamine/Diphenylamine	169	168, 167

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Pentachlorobenzene	**250**	**252, 108, 248, 215, 254**
Pentachloronitrobenzene	**237**	**142, 214, 249, 295, 265**
Pentachlorophenol	**266**	**264, 268**
Phenanthrene	**178**	**179, 176**
Phenol	**94**	**65, 66**
Pyrene	**202**	**200, 203**
Pyridine	**79**	**52**
1,2,4,5-Tetrachlorobenzene	**216**	**214, 179, 108, 143, 218**
2,3,4,6-Tetrachlorophenol	**232**	**131, 230, 166, 234, 168**
m-Toluidine	**106**	**107, 79**
1,2,4-Trichlorobenzene	**180**	**182, 145**
2,4,5-Trichlorophenol	**196**	**198, 97, 132, 99**
2,4,6-Trichlorophenol	**196**	**198, 200**
Acenaphthene-d_{10} (IS)	**164**	**162, 160**
Chrysene-d_{12} (IS)	**240**	**120, 236**
1,4-Dichlorobenzene-d_4 (IS)	**152**	**150, 115**
Naphthalene-d_8 (IS)	**136**	**68**
Perylene-d_{12} (IS)	**264**	**260, 265**
Phenanthrene-d_{10} (IS)	**188**	**94, 80**
2-Fluorobiphenyl (Surrogate)	**172**	**171**
2-Fluoroophenol (Surrogate)	**112**	**64**
Nitrobenzene-d_5 (Surrogate)	**82**	**128, 54**
Phenol-d_6 (Surrogate)	**99**	**42, 71**
Terphenyl-d_{14} (Surrogate)	**244**	**122, 212**
2,4,6-Tribromophenol (Surrogate)	**330**	**332, 141**

TABLE 6

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34
Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34
1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

*** Note**: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
Pyridine	Nitrobenzene	1,2,4,5-Tetrachlorobenzene	2,4,6-Tribromophenol, surr	p-Dimethylamino-azobenzene	Benzo(b)fluoranthene
N-Nitrosodimethylamine	a-,a-Dimethylphenethylamine	Biphenyl	Pentachlorophenol	Chlorobenzilate	Benzo(k)fluoranthene
2-Picoline	Naphthalene	Dimethyl phthalate	Phenanthrene	3,3'-Dichlorobenzidine	Benzo(e)pyrene
Methyl methanesulfonate	4-Chloroaniline	3-Nitroaniline	Anthracene	Benzo(a)Anthracene	Benzo(a)pyrene
2-Fluorophenol, surr	2,6-Dichlorophenol	Acenaphthene	Carbazole	Chrysene	Perylene
Ethyl methanesulfonate	Hexachloropropene	2,4-Dinitrophenol	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	3-Methylcholanthrene
Phenol	Hexachlorobutadiene	Dibenzofuran	Isodrin	Di-n-octylphthalate	Indeno(1,2,3-cd)pyrene
Aniline	N-Nitroso-di-n-butylamine	4-Nitrophenol	Fluoranthene	7,12-Dimethylbenz(a) anthracene	Dibenzo(a,h)anthracene
Phenol-d6, surr	4-Chloro-3-Methylphenol	Pentachlorobenzene	Benzidine		Benzo(g,h,i)perylene
Bis(2-chloroethyl)ether	2-Methylnaphthalene	2,4-Dinitrotoluene	Pyrene		
2-Chlorophenol	1-Methylnapthalene	1-Naphtylamine	Terphenyl-d14, surr		
1,3-Dichlorobenzene	Hexachlorocyclo-pentadiene	2-Napthylamine	Benzyl butyl phthalate		
1,4-Dichlorobenzene	2,4,5-Trichlorphenol	Fluorene			
Benzyl chloride	2,4,6-Trichlorophenol	Diethyl phthalate			
Benzyl Alcohol	2-Fluorobiphenyl, surr	4-Chlorophenyl-phenylether			
1,2-Dichlorobenzene	2-Chloronaphthalene	4-Nitroaniline			
2-Methylphenol	1-Chloronaphthalene	4,6-Dinitro-2-methylphe			
bis(2-Chloroisopropyl)ether	2-Nitroaniline	N-Nitrosodiphenylamine/ Diphenylamine			
3-Methylphenol/4-ethyl	2,6-Dinitrotoluene	Azobenzene			
N-Nitrosodi-n-propylamine	1,3-Dinitrobenzene	4-Bromophenyl-phenylether			
Hexachloroethane	Acenaphthylene	Phenacetin			
Acetophenone	Benzoic Acid	Hexachlorobenzene			
Nitrobenzene-d5, surr	2,4-Dichlorophenol	Dimethoate			
N-Nitrosopiperidine	1,2,4-Trichlorobenzene	4-Aminobiphenyl			
Isophorone		Pronamide			
2-Nitrophenol		Pentachloronitrobenzene			
2,4-Dimethylphenol		1.3-Dinitrobenzene			
Bis(2-chloroethoxy)methane		2,3,4,6-Tetrachlorophenol			

Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010

1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Determination of Volatile Organic Compounds in Ambient Air Using Specially-Prepared Canisters and Analyzed by GC/MS

References: **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition.** U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

Method TO-15: Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

NJDEP – SRP Low Level USEPA TO-15 Method (NJDEP-LL TO-15 3/2007) March 2009 Revision

1. Scope and Application

Matrices: Ambient Air, Soil Gas

Definitions: Refer to Section 16 and Alpha Analytical Quality Systems Manual

This SOP describes the procedure for the analysis of volatile organic compounds (VOCS) in ambient air. The whole air samples are collected in fused-silica lined (FSL) stainless steel canisters, or Tedlar® bags. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass selective detector (MSD).

The organic compounds that are amenable to this method are listed in Table 9. Other compounds may also be amenable provided they meet the QA/QC requirements of the method.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

This SOP contains addendums for various state-specific requirements. The criteria in these addenda (Addendum C-F) must be adhered to for projects conducted under the state programs.

2. Summary of Method

Samples are collected in precleaned, evacuated FSL canisters or Tedlar® bags.

Samples are pre-concentrated using the Entech 7100A Cryogenic Concentrator. A specified volume of sample is pulled using a vacuum pump through a mass flow controller. The sample is cryogenically concentrated to a volume of less than one mL on a Tenax® trap.

Following pre-concentration, the sample is refocused on the GC transfer line. This step further reduces the sample volume to less than one microliter for injection.

The sample is then injected into the GC, which is used to separate the compounds of interest. All compounds are detected using an MSD.

2.1 Method Modifications from Reference

Initial Calibration modifications: If a target analyte cannot meet the %RSD criteria for relative response factor calibration, then linear regression may be used. A minimum of five calibration points must be incorporated and a correlation coefficient of 0.995 or greater must be achieved. The calibration plot must be printed and approval by a supervisor must be obtained prior to calibration acceptance. If any compound is calibrated using linear regression then after the ICV and prior to any sample analysis, a low point standard must be analyzed to confirm there is no bias resulting from the linear regression calibration used. Recovery of the low point standard must be 60-140% using the linear regression curve.

Continuing calibration and laboratory controlled spike (LCS) modifications: The recoveries of all analytes must be within 70% to 130% of the true value. If more than 10% of the compounds fail these criteria, or if one compound has a recovery less than 50% or greater than 150% the LCS must be re-analyzed. If failure occurs a second time, the instrument must be re-calibrated. Recoveries greater than 150% may be acceptable, provided analytes are not detected in the samples.

Sample Duplicate modifications: Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte detected in one of the analysis at >5x the reporting limit, and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analysis, the RPD is NC.

Section 8.4.1.2 of the TO-15 method requires all canisters to be leak checked for a period of 24 hr via pressurization of the canister. The laboratory conducts the leak check by measuring the vacuum of the canister after a minimum of a 24 hr. period has elapsed, not by pressurizing the canister as per the method.

The % RSD for any analyte must be < 30%, as outlined in Section 10.2.2.7 of this SOP.

Humidified nitrogen is used in place of zero air due to the frequency of detection of VOCs in zero air, particularly at SIM detection limits.

There is no NIST-traceable second source standard currently available for the analytes listed in Table 1, Table 3B, and Table A-7.

3. Reporting Limits

Table 9 lists target analytes and Reported Detection Limit information.

4. Interferences

4.1 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2 System carryover can be a potential problem, particularly for the heavier molecular weight hydrocarbons. Carryover can occur after the analysis of standards or high-level samples. Measures that must be taken to remove this contamination can include the analysis of multiple blanks, lab air, and the purging of the autosampler with nitrogen.

4.3 High moisture content, methane levels and/or carbon dioxide levels may interfere with the chromatography and trapping of target analytes. Dilutions may be performed on these samples; however, the reporting limits will then be elevated.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

All employees performing laboratory procedures must have read and understood the Alpha Analytical Chemical Hygiene Plan. All laboratory procedures must be performed in accordance with the provisions and policies of the manual. All accidents, injuries, spills, or unsafe conditions must be reported immediately to the laboratory manager, and such occurrences must be thoroughly documented.

The analyst must wear a lab coat, gloves, and safety glasses while preparing solutions or handling samples.

Preparation of liquid standards must be performed under a properly functioning fume hood. Preparation and venting of gaseous standards must also be performed under a properly functioning fume hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

6.1.1 FSL canister samples can be collected as grab samples or as time-integrated samples. Time-integrated samples can be collected for a maximum of 12 hours using 2.7-liter canisters, or a maximum of 24 hours to 7 days using 6-liter canisters. One liter canisters are typically used for soil vapor sampling with a sampling flowrate of 100-200 ml/min.

6.1.1.1 Grab samples are collected by opening the canister valve and allowing the canister to fill to ambient pressure. This process takes approximately one minute.

6.1.1.2 Time-integrated samples require the use of a properly calibrated flow controller. The flow controller, if provided by Alpha, is calibrated prior to sample collection and is documented in the Alpha ACS LIMs (Refer to Alpha SOP # 2190 for Canister and Flow Controller Preparation).

6.1.2 Tedlar® bag samples typically can be collected as grab or composite samples and may require a pumping system or evacuated box.

6.1.3 Upon receipt at the laboratory, all samples are assigned unique laboratory identification numbers, checked for possible discrepancies, etc. (See SOP # 1559.)

6.2 Sample Preservation

Canisters-None. Tedlar® bags-should be protected from light.

6.3 Sample Shipping

All samples must be accompanied by a chain of custody form, which documents the date, and time of sample collection.

6.4 Sample Handling

The pressure of all FSL canister samples is measured upon receipt at the laboratory and documented in the ACS LIMs (See Alpha SOP #2190). A pressure gauge is attached to the canister inlet, the canister valve is briefly opened and the pressure is recorded. The gauge apparatus used to measure ambient air samples must be separate from that used to measure soil vapor or other matrices known to have elevated levels of VOCs to avoid cross-contamination.

Samples with pressures greater than -15 inches Hg are considered acceptable for analysis.

Samples with less than -15 inches Hg should be pressurized to > -15 inches Hg in order for the concentrator system to accurately draw the correct volume, resulting in a dilution of the sample. For ambient air samples, the client must be notified prior to sample analysis since this dilution may cause reporting limits to be elevated above project action levels.

Any samples that undergo pressurization prior to analysis are documented in the instrument software. Refer to Section 10.3.3.6 for the calculation of dilution factors due to pressurization of samples.

Refer to SOP # 1559 for Sample Management information.

FSL canister and Tedlar® bag samples are stored in the Volatiles Laboratory until analysis has been completed. Tedlar® bag samples are stored in opaque containers.

The recommended holding time for the analysis of FSL canister samples for TO-15 is 30 days. The recommended holding time for the analysis of Tedlar® bag samples for TO-15 is 48-72 hours. Tedlar® bag samples requiring TO-15 analysis may be transferred into canisters upon receipt at the laboratory in order to extend the holding time of the sample to 30 days.

Samples designated by client to be held for subsequent analyses or are “on hold” are to be kept in a designated area in the laboratory. “Hold” samples are discarded upon client authorization or after holding time expiration date.

7. Equipment and Supplies

7.1 Microliter syringes: 10, 25,and 500 µL

7.2 Gas tight syringes: 1 mL, 5 mL, 25 mL, 50 mL, and 100 mL

7.3 FSL canisters: 1.0, 2.7, 6.0 and 15 Liter

7.4 Tedlar® bags: Various sizes. Alpha supplies 5-Liter sizes. All bags must have polypropylene fittings which are recommended for the analysis of Sulfides and Mercaptans (see App. A).

7.5 Stop watch

7.6 Sample Concentrator

7.6.1 The concentrator system consists of two separate pieces of equipment: (1) Entech Model 7016CA VOC Autosampler, and (2) Entech Model 7100A Cryogenic Concentrator using liquid nitrogen.

7.6.2 A vacuum pump (Vaccubrand Model ME2) delivers the sample from the autosampler to the cryogenic concentrator FSL-lined steel tubing.

7.7 Gas Chromatograph System

7.7.1 Gas chromatograph - Hewlett Packard Model 6890N GC or equivalent

7.7.2 Chromatographic column: Restek RTX-1; 60 meters, 0.25 mm ID, 1 micron film thickness

7.7.3 Transfer line from column to GC injection port: Hydroguard ™ 0.32 mm capillary tubing connected to column with Restek Vu-Union connector.

7.8 Mass Spectrometer System

7.8.1 Mass spectrometer - Agilent Models 5973, 5975 or 5977.

7.8.2 The mass spectrometer must be capable of scanning from 29 to 270 amu every 3 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum that meets all the criteria in Table 5 when 50 ng of 4- bromofluorobenzene is injected. For SIM (selective ion monitoring) analysis, the system must be capable of simultaneous SIM/full scan acquisition.

7.8.3 Data System - EnviroQuant ChemStation G1701 DA Version D.02.00 SPI or later for data acquisition and version E.02.00 for data processing.

7.9 Dynamic Diluter

7.9.1 Entech 4600A Dynamic Dilution System-for preparing calibration standards in canisters and performing sample dilutions in canisters.

7.10 Primary flow measurement device: BIOS Cell Defender 510 or equivalent

8. Reagents and Standards

8.1 DI Water or Carbon-filtered tap water

8.2 High purity purge and trap grade methanol (Fisher part # A453-1 or equivalent) for MS source cleaning

8.3 Ultra high purity (UHP) helium for the GC/MS system

8.4 Ultra high purity (UHP) nitrogen for standard preparation

8.5 NIST certified TO-15 gas standards, purchased from Linde (formerly Spectra Gases). Standards are stored at room temperature and expire one year from production date, unless re-certified.

8.6 Neat chemicals: Listed in Table 1, Table A-1, and Table 3B, $\geq$ 98% purity.

8.7 Liquid nitrogen: For the concentrator system and/or GC cooling

8.8 Primary Standards

8.8.1 Primary standard mixtures of TO-15 analytes are purchased certified gaseous standards already prepared as well as gaseous standards prepared in the laboratory by injecting neat chemicals into Tedlar® bags (See Table 1).

8.8.2 Table 1 indicates volumes of neat chemicals that are injected into 20 L of zero air or UHP nitrogen to obtain primary standard concentrations for all analytes.

8.8.3 Purchased primary standards are assigned a CSS # (commercially supplied standard) upon receipt for tracking purposes. Preparation of primary standards must be entered into the primary standard preparation logbook (Form No.: 117-11).

8.8.4 Standards are valid per the manufacturer's expiration date as noted.

8.9 Secondary Standards

8.9.1 Prepare secondary standards in canisters using the Entech 4600A Dynamic Diluter at a minimum of two concentration levels. Table 3A and 3B outlines the preparation steps for each secondary standard.

8.9.2 Prior to preparation of the standards, verify that an appropriate vacuum exists in the canister (>0.5 psia). Figure 3 demonstrates the standard preparation system.

Primary standards prepared in Tedlar bags are injected into a canister (typically 15 L) using an injection tee with a septum or transferred from purchased cylinders via the dynamic diluter.

8.9.3 Attach the transfer lines from the primary standards to the back of the dynamic diluter.

8.9.4 Prior to the injection of the gaseous standards, allow the dynamic diluter to equilibrate for a minimum of 60 minutes by allowing the diluent gas to flow at a rate of 200 mL/min and the primary standard to flow at the rate specified in Table 3A for the appropriate standard being prepared.The flow rate settings are adjusted via the 4600A software, the diluent gas flows through MFC 1 and the calibration standard flows through MFC 2, 3 and 4. Be sure that the isolation valve is open while equilibrating and the vent line is attached to the outlet.

8.9.5 After equilibrating the system, close the isolation valve and attach the canister to the outlet of the diluter. Set stopwatch or timer to the duration specified in Table 3 for the purchased cylinder primary standards. Open the isolation valve and the canister valve and start the timer.

Equation 1: Flow rate calculation:

$$T_f = V_{std} / F_{std}$$

Where:

T_f = standard transfer time, minutes
V_{std} = standard volume, mL
F_{std} = standard flowrate, mL/min

8.9.6 Inject the appropriate amount of Tedlar bag primary standard and the low vapor pressure compounds listed in Table 3B into the injection port tee. This injection must be done while the canister is below atmospheric pressure.

8.9.7 When all the primary standards have been added to the canister, pressurize the canister to 30 psia with humidified nitrogen using the dynamic diluter. This is equivalent to 30 liters of calibration standard in the canister when a 15 L canister is utilized for standard preparation.

NOTE: Standard canisters prepared for analysis using the autosampler must have a maximum pressure of 30 psia to ensure proper and consistent sampling by the instrument.

8.9.8 Label the canister accordingly and record the standard preparation in the secondary standard (SS) preparation logbook (Form No.: 117-12).

8.9.9 The ICV/LCS standard is prepared in the same manner, using primary standards of differing lot #s, at a concentration of 10 ppbV

8.9.10 Standards are valid for 3 months

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8.10 Internal Standard and BFB Tuning Standard/Surrogate Standard

An internal standard (Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-D5) and tuning/surrogate standard containing Toluene-D8, 1,2-Dichloroethane-D4, and Bromofluorobenzene (BFB) are purchased as two separate gas standards at 25 ppbV (2 year expiration date). The internal standards and BFB / surrogates are loaded onto the sample trap prior to the sample via a mass flow controller. The concentration of the internal standard and BFB / surrogates added is based upon the nominal concentration of sample that is analyzed. If the nominal volume of sample is 250 mL, then 100 mL of the 25 ppbV internal standard mix will yield a true value of 10 ppbV for the internal standards and BFB. Using equation 7, the ug/m^3 equivalent of BFB injected 179 ug/m^3 or 179 ng/L (MW of BFB = 175). Thus, the total ng injected is:

$$\text{Total ng} = 179 \text{ ng/L} \times 0.100 \text{ L} = 17.9 \text{ ng}$$

8.11 Instrument Calibration Standards

Calibration standards are prepared by injecting different volumes of the secondary standards into the concentrator/GC/MS system. The low standard will be used to establish the reporting limit for sample analyses. These are described in more detail in Section 10.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

At a minimum, for each day of analysis, a Continuing Calibration standard, Laboratory Method Blank, Laboratory Control Spike and Laboratory Duplicate must be analyzed. Laboratory Control Spike Duplicate (LCSD) will be analyzed only upon client request.

9.1 Laboratory Method Blank(s)

A FSL canister pressurized with humidified nitrogen is utilized as the Laboratory Method Blank. This method blank must be free of target analyte contamination at or above the reporting limit. If it is not, the system must be evaluated for possible sources of contamination. Once the source is determined and eliminated, the Blank must be reanalyzed.

A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred. If samples have been analyzed using an autosampler, data must be evaluated for potential carryover and re-analyses conducted, as appropriate.

9.2 Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)

NOTE: A Laboratory Control Spike Duplicate is only performed when specified by the project requirements and/or upon Client request.

Laboratory Control Spike - A Laboratory Control Spike (LCS) is prepared by spiking an evacuated FSL canister with a different primary standard solution than that used for the calibration or a purchased gaseous standard with the components of interest may be used. If the recovery is not within acceptance criteria, the LCS may be analyzed a second time. If the LCS failure continues, the instrument must be recalibrated. Refer to Section 12 for appropriate corrective actions to be taken. QC limits are subject to change for any particular analyte, if deemed necessary after review of QC control limits.

9.3 Initial Calibration Verification (ICV)

A mid-range calibration standard must be analyzed after the initial calibration and prior to sample analysis, and must be a different source than that used for the initial calibration. See section 10.4.2.5 for additional criteria. Otherwise, sample analysis may proceed.

9.4 Continuing Calibration Verification (CCV)

A mid-range calibration standard must be analyzed prior to sample analysis. This standard is of a different source than that used for the LCS/LCSD pair, typically the same source as the initial calibration standards. See section 10.4.2.5 for additional criteria. If repeated failure of the CCV occurs, the instrument must be recalibrated. Otherwise, sample analysis may proceed. The LCS may also be utilized as the continuing calibration unless otherwise defined in an addendum.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

1) A Laboratory Duplicate is a replicate analysis of a sample. The RPD of duplicate analyses must not exceed 25. Up to 10% of the target analyte detections may exceed acceptance criteria. The criteria does not need to be applied to concentrations less than 5X the reporting limit. If more variation occurs, the sample analysis must be repeated. If an analyte is detected in one analysis at >5x the reporting limit and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analyses, the RPD is NC. Equation 9 is used to calculate the RPD.
The sample chosen for duplicate analysis should not be a trip blank, field blank, or equipment blank. The sample chosen for duplicate analysis must be rotated among clients and/or sites. If possible, field duplicates should not be chosen for duplicate analysis, nor should outside air samples if indoor air samples are also included in the analytical batch.

Equation 9: RPD Calculation

$$\mathbf{RPD = ABS(C_s - C_d) / [(C_s + C_d)/2]*100}$$

where:

RPD = relative percent difference
C_s = concentration in original sample analysis
C_d = concentration in duplicate sample analysis

9.7 Method-specific Quality Control Samples

9.7.1 BFB Tune - A successful BFB spectrum must meet the criteria in Table 5 prior to sample analysis. If a successful BFB spectrum is not obtained, the MS must be retuned and the BFB spectrum re-evaluated prior to analyzing samples.

9.7.2 Internal Standards - The internal standard area counts of each sample, blank, and Laboratory Control Sample are evaluated against the corresponding continuing calibration standard. The internal standard area counts must be within 60-140% of the continuing calibration standard area counts. If The retention times of the internal standards must be within +/- 0.33 min. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Sample must be reanalyzed. In addition, area counts for internal standards for continuing calibration must be within 60-140% recovery of initial calibration. Refer to Sect. 12 for contingencies on samples exhibiting internal standard recovery failures.

9.7.3 TIC Internal Standards - Internal standards used for the quantitation of TICs must be evaluated by comparing the total ion area counts of the internal standards in the samples to the total ion area counts of the internal standards in the blanks. The internal standard area counts must be within 50-200% of the blank area counts. If the internal standards fall outside this range, a different internal standard or an estimated internal standard total ion area must be used to quantitate the TIC. This estimate can be done by using the total ion area from a blank or a clean sample within the analytical batch.

9.8 Method Sequence

- BFB Tune Check
- Calibration Standards (initial) or Continuing Calibration
- Laboratory Control Sample (may be used as the ICV or CC)
- Laboratory Control Sample Duplicate (if needed)
- Laboratory Method Blank
- Samples
- Laboratory Duplicate

Injections may be made until 24 hours after the injection used to check the BFB tune.

All analytical sequences must be recorded in the instrument software and documented in the instrument logbook (Form 117-09)

10. Procedure

10.1 Equipment Set-up

10.1.1 Canister Cleaning and Certification

Refer to Alpha SOP #2190 for canister and flow controller preparation.

10.1.2 Sample Preparation and Concentration

Ensure the integrity of the canister sample as described in Section 6.4. General description of procedure: A 3-stage concentration technique called Cold-Trap Dehydration is used to analyze VOC's in air. The air sample is first concentrated to about a 0.5cc volume by drawing an aliquot of sample simultaneously through a cold trap (no packing material) and then through a Tenax trap. The cold trap is then heated to 10 ^{o}C and is held there while slowly passing helium through it to transfer these compounds to a the Tenax trap, leaving most of the moisture in the cold trap. Sweeping the VOC's from the first to the second trap with only 20cc of helium results in a transfer of less than 0.5 µL of water (40 mL @ 100% RH @ 10 ^{o}C) which can be easily handled by benchtop mass spectrometers. The 20 cc transfer volume also serves to flush the CO_2 through the Tenax trap. After transfer to the second trap, the VOC's are back-flushed while heating to be further focused on an open-tubular focusing trap (cryofocuser) for rapid injection onto the analytical column. Internal standard is added directly to the first stage cryogenic trap prior to the sample by a mass flow controller (MFC). MFC controlled introduction is advantageous over loop injection as it remains consistent with the mechanism used to measure the sample volume.

Connect the canisters or Tedlar® bag(s) to the Entech 7016CA Autosampler. For FSL canisters: Align the tubing from one of the 16 positions to the canister inlet position. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves must be closed at this point. For Tedlar® bags: Connect the valve of the Tedlar® bag with the autosampler line using an adapter fitting.

For canister samples, leak check all inlet connections using the leak check procedure included with the Entech software. A report will be generated indicating the change in vacuum over a period of 30 sec. The vacuum must not increase more than 2 psia. Analysis cannot begin until the leak check has passed for each canister being tested and/or the source of the leak has been determined..

Open the canister or bag valves.

Set up the sequence of the Entech system to withdraw 250 mL from each sample. If high concentrations are expected, lower volumes can be used (minimum of 25 mL).. Samples suspected to contain elevated concentrations (i.e. soil vapor, sub-slab, landfill gas) should be pre-screened prior to analysis to obtain more precise dilution information. If screening results indicate elevated concentrations of non-target analytes, the sample should be diluted such that the peak height of the non-target analyte is approximately 10X greater than the peak height of the first internal standard. Recommended concentrator operating parameters are provided in Table 6.

10.2 Initial Calibration

10.2.1 GC Conditions

Oven program: 25° C, hold for 5.0 minutes, then:

Ramp 1: 100° C at 8.0^{o} C / min.; hold for 0.0 min

Ramp 2: 220° C at 25° C / min.; hold for 4.0 min

Gas Flows

Helium carrier gas flow program: 2.0 mL/min for entire run (23.18 min)

Sample Injection

Injection mode:	split
Injection port temperature:	250^{0} C
Inlet pressure:	27.3 psi
Total flow:	39.3 mL/min
Split ratio:	17.3
Split flow:	34.6 mL/min
Gas saver flow:	OFF

10.2.2 MS Conditions

Temperature of MSD transfer line: 250^{0} C

Temperature of MS Quad: 150^{0} C

Temperature of MS Source: 230^{0} C

Solvent Delay: 3.0 minutes

Scanning Parameters: 29-270 amu until 10 min, scan rate = 5.52 scans/sec; then 35-270 amu, scan rate = 3.1 scans/sec. Threshold = 150. Sampling rate = 2. EM offset-variable to achieve response of 200K area counts (+/- 25K) for the internal standard bromochloromethane.

10.2.3 Daily GC/MS Performance Check

10.2.3.1 The first analysis of the day is typically a tune evaluation. The GC/MS system is checked to confirm that acceptable performance criteria for bromofluorobenzene (BFB), which is in surrogate mixture, are achieved. These criteria must be met prior to analyzing further standards, blanks and samples.

10.2.3.2 A maximum injection of 50 ng must successfully meet the BFB spectrum criteria in Table 5.

10.2.3.3 If the spectrum of BFB does not meet the above stated criteria, the analysis must be repeated. If the spectrum of BFB still does not meet these criteria, the GC/MS instrument must be re-tuned.

10.2.3.4 The Daily GC/MS Performance Check must be analyzed every 24 hours or less.

10.2.4 Initial Calibration

10.2.4.1 Analyze a minimum of five different levels by analyzing various volumes of the secondary standards prepared in Table 3 (Table A-3 for sulfide/mercaptan analysis). The lowest standard will be at or below the reporting limit. If the response is not linear at the lowest level for the higher molecular weight compounds, this point must not be included in the calibration curve for these compounds. As a result, the analysis of more than five levels may be required in order to ensure a minimum of five calibration points for each analyte.

Table 4 lists the calibration standard levels and the volumes of the secondary standards needed to achieve these levels.

10.2.4.2 The true value of each of these calibration points is determined by applying a dilution factor that is based on the volume of sample extracted from the canister for each calibration point. Assuming that a volume of 250 mL will be the maximum volume extracted from the samples; this will be the “1X” volume. A dilution factor can be calculated using Equation 2.

Equation 2: Calculation of Instrument Dilution Factor

$$\mathbf{DF = V_{1X} / V_{actual}}$$

where:

DF = dilution factor
V_{1X} = maximum volume sampled, mL
V_{actual} = actual volume sampled for samples and standards

10.2.4.3 Analyze each calibration standard according to the procedures specified in Section 10. The true value of each calibration point is determined by dividing the concentration of the canister by the dilution factor determined using Equation 2.

10.2.4.4 Tabulate the area response of the characteristic ions against the amount for each analyte and internal standard and calculate relative response factors (RRF) for each compound using Equation 3. Perform this calculation for each calibration standard.

Equation 3: Relative Response Factor for Individual Target Analytes

$$\mathbf{RRF = [(A_{EC}) * (C_i)] / [(A_{EI}) * (C_c)]}$$

where:

RRF = relative response factor
A_{EC} = area count of the extracted ion for the analyte of interest
C_i= amount of internal standard (ppbV)
A_{EI} = area count of the extracted ion for the associated internal standard
C_c = amount of analyte of interest (ppbV)

Table 7 lists all TO-15 analytes, internal standards and the associated quantitation ion.

Table 8 lists the internal standards and the associated TO-15 analytes.

10.2.4.5 Calculate the average response factor for each of the target analytes by the following equation (AVG_x = SUM(RFs) / total # of RFs).

10.2.4.6 Calculate the percent relative standard deviation (%RSD) of the response factors over the secondary range of the curve for each of the target analytes using Equation 4.

Equation 4: Percent Relative Standard Deviation

$$\mathbf{\%RSD = [(SD_n - 1) / (AVG_x)] * 100]}$$

where:

%RSD = percent relative standard deviation
SD_n -1 = standard deviation (n-1 degrees of freedom)
AVG_x = average response factor from the initial calibration curve

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

10.2.4.7 If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for two (2) of the analytes to be above 30%, but below 40% RSD (applies to analytes flagged with a “C” on the Enviroquant initial calibration summary table).

Alpha, may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality

assurance project planners, and also for analytes not listed in EPA Method TO-15:

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when analyzing samples using this calibration.

Calibration points may be removed from the calibration curve to meet the 30% RSD criteria, so long as five consecutive points remain in the calibration curve, and the following procedure is followed:

- Remove high level calibration points
- Remove low-level calibration points; reporting limits will need to be elevated, however.

10.2.4.8 If calibration points in the mid-level range need to be removed due to a sequence error or instrument malfunction, the entire calibration level must be removed from the calibration curveIf the %RSD >30, a calibration curve is generated using the EnviroQuant quantitation software. The correlation coefficient (linear) for the calibration curve must be greater than 0.995. If these criteria cannot be met, prepare a new set of calibration standards and recalibrate the instrument. NOTE: Quadratic calibration in any form is not acceptable.

Authorization from the department supervisor is required prior to using linear regression calibration. Linear regression is only allowed if certain criteria listed below are met:

- The minimum number of points for a linear regression curve is five points.
- The curve must be plotted and printed and turned in with the raw data.
- A calibration standard must be analyzed at the low point of the curve. Recovery of the low point standard must be 60-140% using the linear regression curve.
- The recovery of the compound for the continuing calibration / LCS must be within 70-130%.

10.2.4.9 The reference spectra for all target analytes are reviewed for both assignments and purity for all instruments. In addition, this process of reviewing all spectra continues whenever a new calibration is completed. Reference spectra should be updated with each initial calibration performed with the midpoint standard of the calibration.

10.2.4.10 Internal Standard Criteria for Initial Calibration Curve

The mean response for each internal standard compound is calculated over the initial calibration range. The area response at each calibration level must be within 60-140% of the midpoint area response over the initial calibration range for each internal standard. If recovery is outside the

range, re-analyze calibration level. This criteria must be met prior to sample analysis.

All of these criteria must be met prior to sample analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 GC/MS ANALYSIS

10.3.1.1 The Entech 7100A Concentrator is programmed to the specific analytical conditions listed in Table 6 (Entech method Alpha_TO15.CTD) and the GC/MS parameters are set to those listed in Sections 10.3.1 and 10.3.2. (Enviroquant method TO15-SFS.M (SIM and full scan) or TO15_FS_35C.M (full scan only, for sulfide & mercaptan analysis in App. A)).

10.3.1.2 The BFB spectrum is evaluated by analyzing a Laboratory Method Blank and adding 100 mL of the BFB/surrogate mix.

10.3.1.3 A continuing calibration and/or a laboratory control spike is analyzed. See sect. 9.2 for acceptable criteria

10.3.1.4 A Laboratory Method Blank is analyzed. The Laboratory Method Blank consists of the analysis of 250 mL from a canister of humidified nitrogen. The method blank must be free of target analyte contamination at or above the reporting limit.

10.3.1.5 A 250-mL aliquot of sample is preconcentrated on the Entech 7100A concentrator and injected onto the GC column. For soil vapor samples, or other samples that may contain elevated levels, the aliquot amount must be determined using the results from a pre-screening analysis.

10.3.1.6 Instrument Dilutions and Sub-Atmospheric Sample dilutions

10.3.1.6.1 For dilutions, smaller sample volumes (<250 mL) are analyzed. The smallest volume that can be analyzed with accuracy using the Entech concentrator is 25 mL. The dilution factor is accounted for by entering the volume analyzed in the sample calculation discussed in Section 10.2.2.2 (Equation 2).

10.3.1.6.2 Samples that arrive at the laboratory with pressures below -15 inches Hg should be pressurized with nitrogen to greater than -15 inches Hg, as discussed in Section 6.4. This pressurization results in a dilution factor. The dilution factor is calculated using Equation 6, and the canister dilution spreadsheet (Form No.: 117-05). Attach a green tag to the canister with the pressurization information (initial pressure and final pressure) recorded on the tag.

Equation 6: Dilution Factor for Pressurization of Subatmospheric Samples: Three Steps

Step 1: Calculate the volume in the canister prior to pressurization (Assume a 2.7-liter canister is used).

$$V_{ci} = 2.7 * P_I / 14.696$$

Step 2: Calculate the volume in the canister after pressurization.

$$V_{cf} = 2.7 * P_F / 14.696$$

Step 3: Calculate the dilution factor.

$$DF = V_{cf} / V_{ci}$$

where:

V_{ci}= volume of air in canister prior to pressurization, L
P_I = pressure reading of canister prior to pressurization (psia)
V_{cf} = volume of air in canister after pressurization, L
P_F = pressure reading of canister after pressurization (psia)
DF = dilution factor
14.696 = atmospheric pressure (psia)

10.3.1.6.3 If samples require larger dilutions than pressurization and instrument dilutions, a syringe dilution into an additional canister or Tedlar bag (typically used only for App. A analytes) with a known volume of nitrogen is required.

10.3.1.6.4 Fit a VCO® adapter with a septa to the pressurized sample canister. With a gastight syringe remove appropriate sample size for dilution. Allow sample to flow through syringe for 1 – 2 seconds to flush syringe prior to volumizing. Inject the sample aliquot into a Tedlar bag. If using an evacuated canister, connect the canister to an injection port tee (see Figure 3) attached to the dynamic diluter. Inject the aliquot of sample while a steady stream of Nitrogen is flowing into the dilution canister. Pressurize this canister to 30 psia. Attach a green tag to the canister with dilution information recorded on the tag. Use the dilution calculation worksheet (Form No.: 117-05) to calculate resulting dilutions.

10.3.2 Qualitative Identifications

10.3.2.1 An analyst competent in the interpretation of mass spectra must identify the target analytes by comparison of the sample mass spectrum to the mass spectrum of the standard. Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.

10.3.2.2 For establishing correspondence of the GC RRT, the RRT of the component in the sample must compare within ± 0.06 RRT units of the RRT of the component in the standard. If co-elution of interfering

components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned using extracted ion current profiles for the ion unique to the component of interest.

10.3.2.3 For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same instrument conditions are required. Reference spectra should be updated for each initial calibration performed, using the mid-level standard. Once obtained, these standard spectra may be used for identification and reference purposes.

10.3.2.4 The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
- The relative intensities of ions specified must agree within ± 20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

Table 7 lists the primary and secondary ions for all analytes.

10.3.2.5 Manual integrations: for peaks that are observed to be not integrated correctly by the quantitation software, manual integrations must be performed. Please refer to the manual integration SOP for further instruction on how to properly perform and document manual integrations (Alpha SOP # 1731).

10.3.2.6 **Tentatively identified compounds (TICs)-**A library search may be performed for non-target sample components for the purpose of tentative identification, as requested by the client. Mass spectra are compared to the National Institute of Standards and Technology Mass Spectral Library (2002 version), and a qualitative match is determined by the analyst. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

10.3.2.7 Guidelines for making tentative identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 20% of the most abundant ion) must be present in the sample spectrum.
- The relative intensities of the major ions must agree within ± 30%.

- Molecular ions present in the reference spectrum must be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.
- If, in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound will be reported as "Unknown". The mass spectral interpretation specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid, unknown chlorinated compound)..

10.4 Continuing Calibration

10.4.1 Calibration Verification

10.4.1.1 The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) sample. (The ICV may also be used to satisfy LCS requirements.) This analysis must be performed every time an initial calibration is performed.

10.4.1.2 The ICV must be prepared using a purchased gaseous standard (from a different lot # or separate vendor) with the components of interest in an evacuated FSL canister. Follow the standard preparation procedure for the calibration standards outlined in Section 8.0. The standard must be prepared at or below the midpoint of the calibration curve.

See section 10.4.2.5 for acceptable criteria.

10.4.2 Continuing Calibration

10.4.2.1 A continuing calibration check must be performed daily prior to sample analysis. The continuing calibration standard must be one of the initial calibration levels.

10.4.2.2 Analyze a calibration standard that is at the midpoint of the calibration curve.

10.4.2.3 The LCS standard may be utilized as the continuing calibration check, provided that all target analytes of interest are present in the LCS standard.

10.4.2.4 Calculate the percent difference (%D) of the continuing calibration response factor from the initial calibration average response factor using Equation 5.

Equation 5: Percent Difference

$$\% D = [(C_{found})-(C_{true}) / (C_{true})] * 100$$

where:

%D = percent difference

C_{found} = amount of the analyte detected in the standard (ppbV)
C_{true} = true amount of the analyte in the standard (ppbV)

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

10.4.2.5 Acceptance Criteria

The acceptance criteria is less than 30% RSD for any analyte, with an allowance of two analytes to be greater than 30%, but less than 40%.

Alpha may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality assurance project planners, and also for analytes not listed in EPA Method TO-15:

If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when analyzing samples using this calibration.

10.5 Preventive Maintenance

Mass flow controllers should be checked annually for flowrate accuracy using a BIOS cell or other primary flow measurement device.

Ion source cleaning – typically prior to initial calibration.

Electron Mulitiplier (EM)-changed when the voltage setting required to achieve adequate response approaches 1900.

Rough pump oil changed annually.

Transfer lines, concentrator traps, and the GC guard column should be changed semi-annually, or when system repeatedly fails initial calibration.

11. Data Evaluation, Calculations and Reporting

11.1 Calculations

11.1.1 Individual Target Analytes: The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample

analyses. Standards are prepared on a ppbV basis, so if no dilution is performed, values can be reported from the quantitation report without any calculations. Dilution factors are calculated using Equation 2. Equation 7 shows the conversion of ppbV to µg/m^3.

Equation 7: Conversion of ppbV to µg/m^3

µg/m^3 = (ppbV) * MW / 24.45

where:

24.45 = molar gas constant (g/g-mole)
MW = molecular weight of the compound of interest (Table 1 and 2 lists the molecular weights of the target analytes)

11.1.2 TICS: An estimated amount for the TIC is calculated using the total area of the TIC, the total area of the internal standard assigned by the quantitation software, and a response factor of 1.000 (Equation 8). If the internal standard assigned by the quantitation software exhibits significant interference from other analytes, the next closest eluting internal standard will be utilized.

Equation 8: Calculation of TIC Results in ug/m^3

ug/m^3 = $[(A_T) * (C_{IS})] / [(A_{IS\text{-}T}) * (1.000)]$

where:

A_T = total ion area of the TIC to be measured

C_{IS} = amount of the internal standard

$A_{IS\text{-}T}$ = total ion area of the closest eluting internal standard

The integration of target analytes and internal standards must be performed from valley to valley.

11.2 Data Package

11.2.1 Canister Cleaning Information

A copy of the data for the batch certification analysis associated with the FSL canisters must be on file. The raw data must include a sample chromatogram, quantitation report, and spectra of all positive results.

11.2.2 BFB Tune Checks

Tune checks must be included for all days of analysis, including initial calibration. Raw data must include the chromatogram, mass spectra, and summary of relative abundances of the BFB ions.

11.2.3 Calibration Data

- Initial calibration summary (including average response factors, %RSDs, and copies of calibration curves, if appropriate) for target analytes and all calibration chromatograms must be on file.
- Continuing calibration summaries (including %Ds) for individual analytes.
- Chromatograms and quantitation reports associated with all standards used, in the initial and continuing calibrations.

11.2.4 QA/QC

- Internal standard responses and % recoveries vs. the continuing calibration.
- Quantitation report and chromatogram for laboratory control spike (and laboratory control spike duplicate, if requested).
- Quantitation reports, chromatograms, and spectra of positive results for all blanks.
- Copy of the instrument runlog.

11.2.5 Sample Data

- Quantitation reports, chromatograms, spectra of positive results, negative proofs, and pre- and post-manual integrations for all LCSs, samples, and duplicates.
- A copy of the canister dilution worksheet, Form No.: 117-05 (if any canister pressurizations or canister dilutions are performed).

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

Holding time exceedence and/or container damage is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV, LCS or LCSD recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate Narratives. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

Samples exhibiting internal standard recovery failures must be re-analyzed at the same dilution level if instrument malfunction is suspected to be the cause, or at a lesser dilution if sample matrix or concentration levels of target and/or non-target analytes are suspected of being the cause. If recovery failures are observed upon re-analysis, narrate exceedances.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP # 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP # 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP # 1732 MDL/LOD/LOQ Generation

SOP # 1739 IDC/DOC Generation

SOP # 1797 Hazardous Waste & Sample Disposal

SOP # 1731 Manual Integration

Form 117-05: Canister Dilution Worksheet Template

Form 117-09: Instrument Run Log

Form 117-11: Primary Standard Preparation Log

Form 117-12: Secondary Standard Preparation Log

16. Attachments

Table 1 TO-15 Tedlar® Bag Stock Standard Preparation

Table 2A TO-15 Purchased Stock Standard Cylinders

Table 2B TO-15 Custom Mix Purchased Stock Standard Cylinder

Table 3A Summary of Working Standard Preparation

Table 3B Preparation of Calibration Standards for Low Vapor Pressure Compounds

Table 4 Calibration Standard Levels

Table 5 BFB Key Ions and Abundance Criteria

Table 6 Entech7100A/7016CA Operating Parameters

Table 7 Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards

Table 8 Internal Standards and the Associated Target Analytes

Table 9A TO-15 Target Analytes and Reporting Limits-Standard List

Table 9B TO-15 Target Analytes and Reporting Limits-Additional Analytes

Figure 3 FSL Canister Standard Preparation System

Appendix A Cold Trap Dehydration technique (CTD) for Analysis of Sulifides and Mercaptans

Appendix B Data Acquisition Parameters and Analysis Modifications for Conducting SIM Analysis

Appendix C Modifications to Data Review and Case Narrative to Comply with MADEP MCP-TO-15 Method

Appendix D Method Modification to Perform the NJDEP TO-15 Low Level Method

Appendix E Modifications to Data Review and Case Narrative to Comply with 2014 NJDEP Technical Guidance for EPA Method TO-15

Appendix F Modifications to Data Review and Case Narrative to Comply with Ohio EPA Voluntary Action Program(VAP) Requirements for EPA Method TO-15

DEFINITIONS

Absolute canister pressure - Pg + Pa, where Pg = gauge pressure in the canister (psig) and Pa = barometric pressure.

Absolute pressure - Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPA, mm Hg, or psia (pounds per square inch absolute).

Cryogen - The refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp = -196^{0}C).

Gauge pressure - Pressure measured above atmospheric pressure (as opposed to absolute pressure). Zero gauge is equal to ambient atmospheric (barometric) pressure. Units = psig (pounds per square inch gauge).

ppmV – parts per million on a volume basis.

ppbV – parts per billion on a volume basis

psia – pounds per square inch absolute

Relative retention time (RRT)– retention time (RT) ratio of the target analyte and the internal standard used to quantitate (RT target / RT internal standard).

Table 1

TO-15 Tedlar® Bag Stock Standard Preparation-Initial Calibration Standard and LCS/ICV Standard

COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected*	FINAL ppmV
Indan	118.18	965	5.0	50.0
Indene	116.16	996	4.8	50.4
1,2,3-Trimethylbenzene	120.19	894	5.5	50.1
Thiophene	84.14	1051	3.3	50.4
2-Ethylthiophene	112.19	990	4.7	50.7
2-Methylthiophene	98.17	1014	4.0	50.6
3-Methylthiophene	98.17	1016	4.0	50.6
Acetone	58.1	791	12.0	200
Acetaldehyde	44.05	785	11.5	250.7
Halothane **	197.38	1872	4.3	49.9
ICV / LCS Standard				
COMPOUND (liquids)	**MOL WGT**	**Density ug/uL**	**uL injected***	**FINAL ppmV**
Acetone	58.1	791	12.0	200
Halothane **	197.38	1872	4.3	49.9

All neat chemicals are injected into a Tedlar® bag containing 20 Liters of zero air or UHP nitrogen.

** Halothane reported via SIM analysis only.

See Table A-1 & Table A-2 for sulfide/mercaptan stock standard preparation

Table 2A

TO-15 Purchased Primary Standard Mix

TO-15 Primary Standard Mix #1					
COMPOUND	**MOL WGT**	**Conc. ppmV**	**COMPOUND**	**MOL WGT**	**Conc. ppmV**
dichlorodifluoromethane	120.92	1.0	cis-1,3-dichloropropene	110.97	1.0
chloromethane	50.49	1.0	trans-1,3-dichloropropene	110.97	1.0
Freon-114	170.92	1.0	1,1,2-trichloroethane	133.41	1.0
vinyl chloride	62.5	1.0	toluene	92.14	1.0
bromomethane	94.94	1.0	1,2-dibromoethane	187.87	1.0
chloroethane	64.52	1.0	tetrachloroethene	165.83	1.0
trichlorofluoromethane	137.37	1.0	chlorobenzene	112.56	1.0
1,1-dichloroethene	96.94	1.0	ethylbenzene	106.17	1.0
methylene chloride	84.93	1.0	m-xylene	106.17	1.0
Freon-113	187.38	1.0	p-xylene	106.17	1.0
trans-1,2-dichloroethene	98.96	1.0	styrene	104.15	1.0
1,1-dichloroethane	96.94	1.0	1,1,2,2-tetrachloroethane	167.85	1.0
cis-1,2-dichloroethene	96.94	1.0	o-xylene	106.17	1.0
chloroform	119.38	1.0	1,3,5-trimethylbenzene	120.2	1.0
1,2-dichloroethane	98.96	1.0	1,2,4-trimethylbenzene	120.2	1.0
1,1,1-trichloroethane	133.41	1.0	1,3-dichlorobenzene	147.0	1.0
benzene	78.11	1.0	1,4-dichlorobenzene	147.0	1.0
carbon tetrachloride	153.82	1.0	1,2-dichlorobenzene	147.0	1.0
1,2-dichloropropane	113	1.0	1,2,4-trichlorobenzene	181.45	1.0
trichloroethene	92.14	1.0	hexachlorobutadiene	260.76	1.0

TO-15 Primary Standard Mix #2		
COMPOUND	**MOL WGT**	**Conc. ppmV**
Propylene	42.08	1.0
1,3-butadiene	54.09	1.0
Vinyl bromide	106.96	1.0
Acetone	58.08	1.0
Isopropyl alcohol	60.1	1.0
Carbon disulfide	76.14	1.0
3-chloropropene	76.53	1.0
Trans-1,2-dichloroethene	96.94	1.0
Methyl-tert butyl ether	88.15	1.0
Vinyl acetate	86.09	1.0
2-butanone (MEK)	72.11	1.0
Hexane	86.18	1.0
Ethyl acetate	88.11	1.0
Tetrahydrofuran	72.11	1.0
Cyclohexane	84.16	1.0
Bromodichloromethane	163.83	1.0
1,4-dioxane	88.11	1.0
2,2,4-trimethylpentane	114.23	1.0
Heptane	100.21	1.0
4-methyl-2-pentanone (MIBK)	100.16	1.0
2-hexanone	100.16	1.0
Dibromochloromethane	208.29	1.0
Bromoform	252.75	1.0
Benzyl chloride	126.59	1.0
4-ethyl toluene	120.2	1.0

All mixes currently purchased from Linde (formerly Spectra Gases)

Table 2B

TO-15 Purchased Custom Mix

TO-15 Custom Standard Mix					
COMPOUND	**MOL WGT**	**Conc. ppmV**	**COMPOUND**	**MOL WGT**	**Conc. ppmV**
Propane	44.10	1.0	n-Octane	114.23	1.0
Chlorodifluoromethane	86.47	1.0	1,1,1,2-Tetrachloroethane	167.85	1.0
Methanol	32.04	5.0	1,2,3-Trichloropropane	147.43	1.0
n-Butane	58.12	1.0	Nonane	128.26	1.0
Dichlorofluoromethane	102.92	1.0	Isopropylbenzene	120.19	1.0
Ethanol	46.07	5.0	Bromobenzene	157.01	1.0
Acetonitrile	41.05	1.0	2-Chlorotoluene	126.58	1.0
Acrolein	56.10	1.0	n-Propylbenzene	120.19	1.0
n-Pentane	72.20	1.0	4-Chlorotoluene	126.58	1.0
Acrylonitrile	53.10	1.0	tert-Butylbenzene	134.20	1.0
Ethyl Ether	74.12	1.0	n-Decane	142.28	1.0
tert-Butyl Alcohol	74.12	1.0	sec-Butylbenzene	134.20	1.0
2,2-Dichloropropane	112.99	1.0	p-Isopropyltoluene	134.22	1.0
Di-Isopropyl Ether	102.17	1.0	n-Butylbenzene	134.20	1.0
Tert-Butyl Ethyl Ether	102.20	1.0	1,2-Dibromo-3-chloropropane	236.33	1.0
1,1-Dichloropropene	110.97	1.0	n-Undecane	156.31	1.0
Tert Amyl Methyl Ether	102.17	1.0	Naphthalene	128.17	1.0
Dibromomethane	173.83	1.0	n-Dodecane	170.33	1.0
1,3-Dichloropropane	112.99	1.0	1,2,3-Trichlorobenzene	181.45	1.0
n-Butyl Acetate	116.16	1.0	Methyl methacrylate	100.12	1.0

All mixes currently purchased from Linde (formerly Spectra Gases)

Table 3A

Summary of Secondary Standards Preparation

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume canister (L)	Final Concentration ppbV
Secondary standards prepared using dynamic dilution system (Entech 4600A)					
Tedlar® bag primary standard	50	60 mL	Syringe Injection	30	100
TO-15 #1 & #2, & Custom Mix	1.0	3000 mL	50 mL/min for 60 min.	30	100
Tedlar® bag primary standard	50	6.0 mL	Syringe Injection	30	10
TO-15 #1 & #2, & Custom Mix	1.0	300 mL	50 mL/min for 6.0 min.	30	10
Secondary standards prepared via serial dilution					
100 ppbV ICAL mix	1.0	300 mL	Syringe Injection	30	1.0
100 ppbV ICAL mix	0.1	30 mL	Syringe Injection	30	0.1

* This calibration standard is used for TO-15 SIM analysis only (see Appendix B).

Table 3B

Preparation of Calibration Standards for Low Vapor Pressure Compounds

COMPOUND (solids)	Vapor Pressure* (P), atm	Molecular Weight	Volume (V) extracted, mL	Gas Constant (R) (L atm/gm mol K)	T, °K	n**	Final Volume, L	mg	ug/m^3	ppbV
1-methylnaphthalene	7.11E-05	142.20	4.2	0.082057	298.1	2.44E-08	30	0.00347	115.7	10
1-methylnaphthalene	7.11E-05	142.00	42	0.082057	298.1	1.22E-07	30	0.0173255	577.5	100
2-methylnaphthalene	8.96E-05	142.00	3.4	0.082057	298.1	2.491E-08	30	0.0035373	117.9	10.2
2-methylnaphthalene	8.96E-05	142.00	34	0.082057	298.1	1.246E-07	30	0.0176867	589.6	101.6
1,2,4,5-tetramethylbenzene	4.23E-04	134.22	2.1	0.082057	291.5	2.473E-08	30	0.0033191	110.6	10
1,2,4,5-tetramethylbenzene	4.23E-04	134.22	21	0.082057	291.5	1.236E-07	30	0.0165954	553.2	101
benzothiophene	7.70E-05	134.20	3.8	0.082057	291.5	2.446E-08	30	0.0032824	109.4	10.0
benzothiophene	7.70E-05	134.20	38	0.082057	291.5	1.223E-07	30	0.016412	547.1	100

Approximately 5.0 g of solid material was allowed to stand in a 250 mL jar w/ septa cap for 30 min prior to removal of vapor phase aliquot. The aliquot was then spiked directly into secondary standard.

All vapor pressure values from Lange's Handbook of Chemistry & Physics w/ the exception of 1,2,4,5-tetramethylbenzene. The initial value was derived using Antoine Equation below, and then normalized using the PIANO vapor phase standard:

Antoine Equation used to calculate vapor pressure

	p, mm Hg	log p	A	B	C	T, C	T, K
1,2,4,5-tetramethylbenzene	0.32108333	-0.4933822	7.08	1672.43	201.43	19.4	292.55

Table 4

Calibration Standard Levels

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.20	50 mL of 1.0 ppbV sec. standard
2	0.50	125 mL of 1.0 ppbV sec. standard
3	1.0	250 mL of 1.0 ppbV sec. standard
4	5.0	125 mL of 10 ppbV sec. standard
5	10	250 mL of 10 ppbV sec. standard
6	20	50 mL of 100 ppbV sec. standard
7	50	125 mL of 100 ppbV sec. standard
8	100	250 mL of 100 ppbV sec. standard

Table 5

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0-40.0 percent of the base peak
75	30.0-66.0 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0-9.0 percent of mass 174
176	Greater than 93.0 percent but less than 101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

Table 6

ENTECH 7016CA/7100A Operating Parameters

Module 1 (Cold Trap)	
Parameter	**Setting**
Trapping Temperature	-40° C
Internal standard / surrogate volume	100 mL
Internal standard / surrogate flow rate	100 mL / min
Nominal Sample volume (may vary depending on sample concentrations)	250 mL
Sample flow rate, mL / min	100 mL / min
Preheat Temperature	10° C
Desorb Temperature	10° C
Bake Temperature	220° C
Bake Time	10 min
Module 1 to Module 2 transfer volume / rate	20 cc @ 5.0 cc/min
Module 2 (Tenax trap)	
Parameter	**Setting**
Trapping Temperature	-65° C to -75° C
Desorb Temperature	220° C
Bake Temperature	220° C
Module 2 to Module 3 desorb time	3.5 min
Module 3 (Cryofocusser)	
Parameter	**Setting**
Cryofocusing Temperature	-130° C to -190° C
Desorb Temperature	Approx. 90° C
Module 3 to GC desorb time	3.0-3.5 min
Bake temperature / event	Approx. 90° C / event 3
Delay time	13-17 min

Table 7
Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards

Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)
bromochloromethane	49	130	1,4-difluorobenzene	114	63	bromoform	173	171, 175
chlorodifluoromethane	51	67	n-hexane	57	43, 86	styrene	104	103, 78
propylene	41	39, 42	diisopropyl ether	87	45, 59	1,1,2,2-tetrachloroethane	83	85
propane	29	43,39	ethyl acetate	61	43, 70	o-xylene	91	106
dichlorodifluoromethane	85	87	2,2-dichloropropane	77	41, 97	1,2,3-trichloropropane	75	39, 110
chloromethane	50	52	tetrahydrofuran	42	71, 72	nonane	43	57, 128
Freon-114	85	87, 135	tert-butyl ethyl ether	59	87, 57	bromofluorobenzene	95	75, 174
methanol	31	32,29	1,2-dichloroethane-D4	65	67, 102	isopropylbenzene	105	120
vinyl chloride	62	64,	1,1,1-trichloroethane	97	61, 119	bromobenzene	77	156
1,3-butadiene	54	39	1,1-dichloropropene	75	39,110	2-chlorotoluene	126	91
butane	43	41,58	benzene	78	52	n-propylbenzene	120	91
bromomethane	94	96	carbon tetrachloride	117	119, 82	4-chlorotoluene	91	126
chloroethane	64	66	cyclohexane	56	84, 41	4-ethyl toluene	105	91, 120
dichlorofluoromethane	67	69, 47	tert-amyl methyl ether	73	43, 87	1,3,5-trimethylbenzene	105	91, 120
ethanol	31	45	dibromomethane	93	95, 174	tert-butlybenzene	119	134
acetonitrile	41	40	1,2-dichloropropane	63	39, 62	1,2,4-trimethylbenzene	105	91, 120
vinyl bromide	106	108	bromodichloromethane	83	85, 129	decane	57	43, 142
acrolein	56	55,29	trichloroethene	130	132, 97	benzyl chloride	91	126
acetone	43	58	1,4-dioxane	88	58	1,3-dichlorobenzene	146	75, 111
trichlorofluoromethane	101	103	2,2,4-trimethylpentane	57	41, 99	1,4-dichlorobenzene	146	75, 111
isopropyl alcohol	45	59	n-heptane	43	57, 100	sec-butylbenzene	105	134
acrylonitrile	53	52,51	cis-1,3-dichloropropene	75	39, 77	p-isopropyltoluene	119	134
pentane	43	57,72	4-methyl-2-pentanone	43	58, 100	1,2-dichlorobenzene	146	75, 111
ethyl ether	31	59,45	methyl methacrylate	41	69, 100	n-butylbenzene	91	134
1,1-dichloroethene	61	96, 63	trans-1,3-dichloropropene	75	39, 77	1,2-dibromo-3-chloropropane	75	39, 157
Tertiary butyl Alcohol	59	41, 43	1,1,2-trichloroethane	97	61, 83	undecane	57	43, 71, 156
methylene chloride	49	84	Thiophene	84	45, 58	dodecane	57	43
3-chloropropene	41	39, 76	chlorobenzene-D5	54	82, 117	1,2,4-trichlorobenzene	180	109, 145
carbon disulfide	76	44	toluene	91	92	naphthalene	128	102
Freon 113	101	85, 151	toluene-D8	98	100	1,2,3-trichlorobenzene	180	109, 145
trans-1,2-dichloroethene	61	96, 98	1,3-dichloropropane	76	41,49	hexachlorobutadiene	225	118, 260
1,1-dichloroethane	63	65	2-hexanone	43	58, 100	2-methylthiophene	97	45, 98
MTBE	73	57, 43	dibromochloromethane	129	127, 131	3-methylthiophene	97	45, 98
vinyl acetate	43	86	1,2-dibromoethane	107	109	2-ethylthiophene	97	45, 112
2-butanone	43	72	butyl acetate	73	43, 56	1,2,3-trimethylbenzene	105	120
cis-1,2-dichloroethene	61	96, 98	octane	85	43, 57, 114	indan	117	91, 118
chloroform	83	85, 47	tetrachloroethene	166	94, 131	indene	115	89, 116
1,2-dichloroethane	62	49, 63, 64	1,1,1,2-tetrachloroethane	131	95, 133	1,2,4,5-tetramethylbenzene	119	91, 134
acetaldehyde	29	43, 44	chlorobenzene	112	77, 114	benzothiophene	134	63, 89
			ethylbenzene	91	106	2-methylnaphthalene	142	115, 141
			m+p-xylene	91	106	1-methylnaphthalene	142	115, 141

Table 8

Internal Standards and the Associated Target Analytes

bromochloromethane		1,4-difluorobenzene	chlorobenzene-D5	
chlorodifluoromethane	trans-1,2-dichloroethene	hexane	toluene	decane
propylene	1,1-dichloroethane	diisopropyl ether	toluene-D8	benzyl chloride
propane	MTBE	tert-butyl ethyl ether	1,3-dichloropropane	1,3-dichlorobenzene
dichlorodifluoromethane	vinyl acetate	1,2-dichloroethane-D4	2-hexanone	1,4-dichlorobenzene
chloromethane	2-butanone	1,1,1-trichloroethane	dibromochloromethane	sec-butylbenzene
Freon-114	cis-1,2-dichloroethene	1,1-dichloropropene	1,2-dibromoethane	p-isopropyltoluene
methanol	chloroform	benzene	butyl acetate	1,2-dichlorobenzene
vinyl chloride	1,2-dichloroethane	carbon tetrachloride	Octane	n-butylbenzene
1,3-butadiene	trans-1,2-dichloroethene	cyclohexane	tetrachloroethene	1,2-dibromo-3-chloropropane
butane	1,1-dichloroethane	tert-amyl methyl ether	1,1,1,2-tetrachloroethane	undecane
bromomethane	acetaldehyde	dibromomethane	Chlorobenzene	dodecane
chloroethane	ethyl acetate	1,2-dichloropropane	Ethylbenzene	1,2,4-trichlorobenzene
dichlorofluoromethane	2,2-dichloropropane	bromodichloromethane	m+p-xylene	naphthalene
ethanol	tetrahydrofuran	trichloroethene	Bromoform	1,2,3-trichlorobenzene
acetonitrile		1,4-dioxane	Styrene	hexachlorobutadiene
vinyl bromide		2,2,4-trimethylpentane	1,1,2,2-tetrachloroethane	2-methylthiophene
acrolein		heptane	o-xylene	3-methylthiophene
acetone		cis-1,3-dichloropropene	1,2,3-trichloropropane	2-ethylthiophene
trichlorofluoromethane		4-methyl-2-pentanone	Nonane	1,2,3-trimethylbenzene
isopropyl alcohol		methyl methacrylate	bromofluorobenzene	indan
acrylonitrile		trans-1,3-dichloropropene	isopropylbenzene	indene
pentane		1,1,2-trichloroethane	bromobenzene	1,2,4,5-tetramethylbenzene
ethyl ether		thiophene	2-chlorotoluene	benzothiophene
1,1-dichloroethene			n-propylbenzene	2-methylnaphthalene
Tertiary butyl Alcohol			4-chlorotoluene	1-methylnaphthalene
methylene chloride			4-ethyl toluene	
3-chloropropene			1,3,5-trimethylbenzene	
carbon disulfide			tert-butlybenzene	
Freon 113			1,2,4-trimethylbenzene	

Table 9A

TO-15 Target Analytes and Reporting Limits
Standard List

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.2	1.09
1,1,2,2-tetrachloroethane	79-34-5	0.2	1.37
1,1,2-trichloroethane	79-00-5	0.2	1.09
1,1-dichloroethane	75-34-3	0.2	0.81
1,1-dichloroethene	75-35-5	0.2	0.79
1,2,4-trichlorobenzene	120-82-1	0.2	1.48
1,2,4-trimethylbenzene	95-63-6	0.2	0.98
1,2-dibromoethane	106-93-4	0.2	1.54
1,2-dichlorobenzene	95-50-1	0.2	1.2
1,2-dichloroethane	107-06-2	0.2	0.81
1,2-dichloropropane	78-87-5	0.2	0.92
1,3,5-trimethylbenzene	108-67-8	0.2	0.98
1,3-butadiene	106-99-0	0.2	0.44
1,3-dichlorobenzene	541-73-1	0.2	1.2
1,4-dichlorobenzene	106-46-7	0.2	1.2
1,4-dioxane	123-91-1	0.2	0.72
2,2,4-trimethylpentane	540-84-1	0.2	0.93
2-butanone	78-93-3	0.5	1.48
2-hexanone	591-78-6	0.2	0.82
3-chloropropene	107-05-1	0.2	0.63
4-Ethyltoluene	622-96-8	0.2	0.98
Acetone	67-64-1	1.0	0.94
benzene	71-43-2	0.2	0.64
Benzyl Chloride	100-44-7	0.2	1.03
bromodichloromethane	75-27-4	0.2	1.34
bromoform	75-25-2	0.2	2.07
bromomethane	74-83-9	0.2	0.78
carbon disulfide	75-15-0	0.2	0.62
carbon tetrachloride	56-23-5	0.2	1.26
chlorobenzene	108-90-7	0.2	0.92
chloroethane	75-00-3	0.2	0.53
chloroform	67-66-3	0.2	0.98

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
chloromethane	74-87-3	0.2	0.41
cis-1,2-dichloroethene	156-59-2	0.2	0.79
cis-1,3-dichloropropene	10061-01-5	0.2	0.91
cyclohexane	110-82-7	0.2	0.69
dibromochloromethane	124-48-1	0.2	1.7
dichlorodifluoromethane	75-71-8	0.2	0.99
ethanol	64-17-5	2.5	4.71
ethyl acetate	141-78-6	0.5	1.8
ethylbenzene	100-41-4	0.2	0.87
Freon-113	76-13-1	0.2	1.53
Freon-114	76-14-2	0.2	1.4
hexachlorobutadiene	87-68-3	0.2	2.13
hexane	110-54-3	0.2	0.7
isopropyl alcohol	67-63-0	0.5	1.23
methylene chloride	75-09-2	0.5	1.74
MIBK	108-10-1	0.5	2.1
MTBE	1634-04-4	0.2	0.72
m+p-xylene	108-38-3 106-42-3	0.4	1.74
n-heptane	142-82-5	0.2	0.82
o-xylene	95-47-6	0.2	0.87
propylene	115-7-1	0.5	0.85
styrene	100-42-5	0.2	0.85
tetrachloroethene	127-18-4	0.2	1.36
tetrahydrofuran	109-99-9	0.5	1.48
toluene	108-88-3	0.2	0.75
trans-1,2-dichloroethene	156-60-5	0.2	0.79
trans-1,3-dichloropropene	10061-02-6	0.2	0.91
trichloroethene	79-01-6	0.2	1.07
trichlorofluoromethane	75-69-4	0.2	1.12
vinyl acetate	108-05-4	0.5	1.75
vinyl bromide	593-60-2	0.2	0.87
vinyl chloride	75-01-4	0.2	0.51

Table 9B

TO-15 Target Analytes and Reporting Limits
Additional Analytes

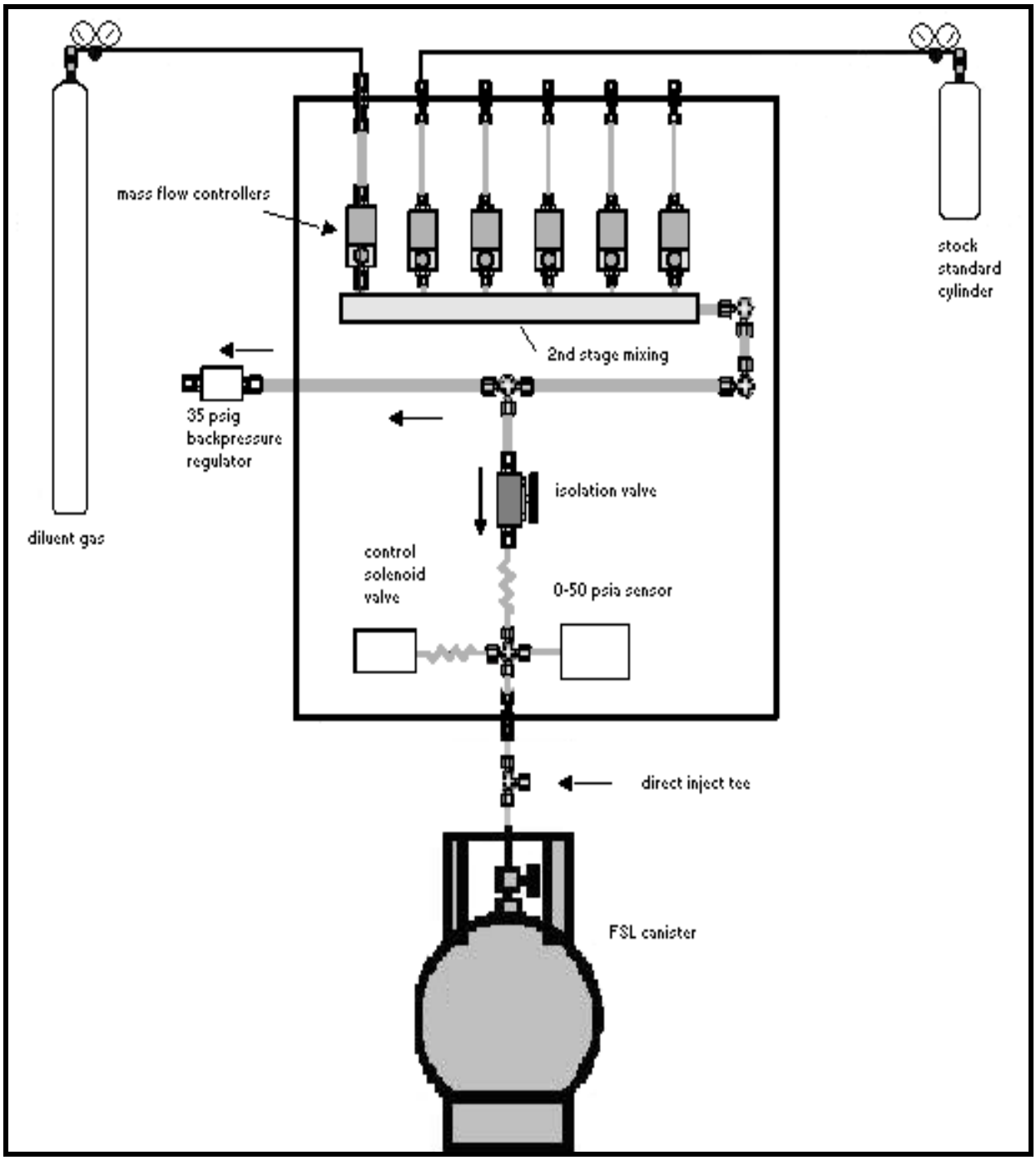
COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
AP-42 Analytes			
acrolein	107-02-8	0.50	1.15
acrylonitrile	107-13-1	0.50	1.08
butane	106-97-8	0.20	0.48
Chlorodifluoromethane	75-45-6	0.20	0.71
Dichlorofluoromethane	75-71-8	0.20	0.84
n-Pentane	109-66-0	0.20	0.59
Propane	74-98-6	0.50	0.90
MADEP MCP 8260 Analytes			
1,1,1,2-tetrachloroethene	630-20-6	0.20	1.37
1,1-dichloropropene	563-58-6	0.20	0.91
1,2,3-trichlorobenzene	87-61-6	0.20	1.48
1,2,3-Trichloropropane	96-18-4	0.20	1.20
1,3-dichloropropane	142-28-9	0.20	0.92
2,2-dichloropropane	594-20-7	0.20	0.92
2-chlorotoluene	95-49-8	0.20	1.03
4-chlorotoluene	106-43-4	0.20	1.03
bromobenzene	108-86-1	0.20	0.79
1,2-dibromo-3-chloropropane	96-12-8	0.20	1.93
dibromomethane	74-95-3	0.20	1.42
diisopropyl ether	108-20-3	0.20	0.84
isopropylbenzene	98-82-8	0.20	0.98
isopropyltoluene	99-87-6	0.20	1.10
naphthalene	91-20-3	0.20	1.05
n-butylbenzene	104-51-8	0.20	1.10
n-propylbenzene	103-65-1	0.20	0.98
sec-butylbenzene	135-98-8	0.20	1.10
tert-amyl methyl ether	994-05-8	0.20	0.84
tert-Butyl ethyl ether	637-92-3	0.20	0.84
tert-butylbenzene	98-06-6	0.20	1.10

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
NYDEC Petroleum Indicator Compounds			
nonane	111-84-2	0.20	1.05
octane	111-65-9	0.20	0.93
undecane	1120-21-4	0.20	1.28
decane	124-18-5	0.20	1.16
dodecane	112-40-3	0.20	1.39
indene	95-13-6	0.20	0.95
Indan	496-11-7	0.20	0.97
thiophene	110-02-1	0.20	0.69
2-methylthiophene	554-13-3	0.20	0.80
3-methylthiophene	616-44-4	0.20	0.80
2-ethyl thiophene	872-55-9	0.20	0.92
benzothiophene	934-80-5	0.50	13.7
1,2,3-trimethylbenzene	526-73-8	0.20	0.98
1,2,4,5-tetramethylbenzene	95-93-2	0.20	1.10
2-methylnaphthalene	91-57-6	1.0	5.8
1-methylnaphthalene	90-12-0	1.0	5.8
Project Specific Analytes			
acetaldehyde	75-07-0	2.5	4.51
Acetonitrile	75-05-8	0.20	0.34
butyl acetate	123-86-4	0.50	0.95
ethyl ether	60-29-7	0.20	0.61
methanol	67-56-1	5.0	6.55
tert-butyl alcohol	75-65-0	0.50	1.25
Methyl methacrylate	80-62-6	0.50	2.05
All reporting limits are subject to change.			

Figure 3

Standard Preparation System

Entech 4600A Dynamic Diluter



Appendix A

Cold Trap Dehydration technique (CTD) for Analysis of Sulfides and Mercaptans by EPA TO-15

Target analytes:

Compound	CAS#	Reporting Limit, ppbV
Hydrogen sulfide	7783-06-4	2.0
Carbonyl sulfide	463-58-1	2.0
Methyl mercaptan	74-93-1	2.0
Ethyl mercaptan	75-08-1	0.5
Dimethyl sulfide	75-18-3	0.5
carbon disulfide	75-15-0	0.5
Isopropyl Mercaptan	75-33-2	0.5
tert-Butyl Mercaptan	75-66-1	0.5
n-Propyl Mercaptan	107-03-9	0.5
Ethyl Methyl Sulfide	624-89-5	0.5

Compound	CAS#	Reporting Limit, ppbV
Thiophene	110-02-1	0.5
Isobutyl Mercaptan	513-44-0	0.5
diethyl sulfide	352-93-2	0.5
Butyl Mercaptan	109-75-5	2.0
dimethyl disulfide	624-92-0	0.5
3-Methylthiophene	616-44-4	0.5
Tetrahydrothiophene	110-01-0	0.5
2-Ethylthiophene	872-55-9	0.5
2,5-Dimethylthiophene	638-02-8	0.5
Diethyl Disulfide	110-81-6	0.5
All reporting limits are subject to change.		

The cold trap dehydration method requires removal of the glass bead trap installed in module 1 of the Entech 7100A concentrator and installing a blank trap (i.e. no trapping material). This trap is cooled and a 250 mL aliquot of sample is allowed to pass through this trap and then directly onto the Tenax trap in module 2, which is also cooled (see Table A-5 for setpoints). The sample is then transferred to module 3 (cryofocusser) via ballistic heating. All requirements stated in this SOP must be applied to any TO15-Sulfide/Mercaptan analysis conducted in the laboratory.

SOP modifications:

Standard preparation and calibration procedures for these analytes are listed in Table A-1 through A-4. Quantitation parameters are listed in Table A-7. Table A-6 lists modified GC conditions.

Section 9.2.5 and 9.5.3.1: Use the Entech method alpha_H2S&SULF.CTD in place of the alpha_TO15.MPT method.

A second source laboratory check standard (LCS) is not readily available for these analytes. An LCS standard is prepared at a different concentration than the initial calibration standards using the same stock standard.

Table A-1

Sulfide/Mercaptan Liquid Cocktail Primary Standard

COMPOUND (liquids)	Initial Conc.	CAS#	Molecular Weight	Density ug/uL	uL injected into cocktail *
ethyl mercaptan	neat	75-08-1	62.14	839	37.0
dimethyl sulfide	neat	75-18-3	62.14	846	36.7
carbon disulfide	neat	75-15-0	76.1	1266	30.1
Isopropyl Mercaptan	neat	75-33-2	76.2	820	46.5
tert-Butyl Mercaptan	neat	75-66-1	90.19	800	56.4
n-Propyl Mercaptan	neat	107-03-9	76.16	841	45.3
Ethyl Methyl Sulfide	neat	624-89-5	76.16	842	45.2
Thiophene	neat	110-02-1	84.1	1051	40.0
Isobutyl Mercaptan	neat	513-44-0	90.19	831	54.3
diethyl sulfide	neat	352-93-2	90.2	837	53.9
Butyl Mercaptan	neat	109-79-5	90.19	842	107.1
dimethyl disulfide	neat	624-92-0	94.2	1046	45.0
3-Methylthiophene	neat	616-44-4	98.17	1016	48.3
Tetrahydrothiophene	neat	110-01-0	88.17	1000	44.1
2-Ethylthiophene	neat	872-55-9	112.19	990	56.7
2,5-Dimethylthiophene	neat	638-02-8	112.19	985	57.0
Diethyl Disulfide	neat	110-81-6	122.25	993	61.6
				Total volume of cocktail mix, uL	**865**

* **Volumes determined using Entech ESP software**

Table A-2

TO15-CTD Tedlar® Bag Stock Standard Preparation

COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected *	FINAL ppmV
Liquid Cocktail Primary Standard (Table A-1)	NA	NA	72	50 / 100 (n-butyl merc)
COMPOUND (gases)	**MOL WGT**	**CONC ppmV**	**mL injected**	**Conc ppmV**
hydrogen sulfide	34.08	1.00E+06	5.0	250.0
carbonyl sulfide	60.08	1.00E+06	5.0	250.0
methyl mercaptan	48.11	1.00E+06	5.0	250.0

*** Volume determined using Entech ESP software**

Table A-3

Summary of Secondary Standards Preparation for Sulfides and Mercaptan Analysis

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume Tedlar® Bag (L)	Final Concentration ppbV
Secondary standards prepared using gas-tight syringes					
Tedlar® bag primary standard-Low	50	0.4 mL	Syringe Injection	4.0	5.0 / 10.0 / 20
Tedlar® bag primary standard-High	50	8.0 mL	Syringe Injection	4.0	100 / 200 / 500
Tedlar® bag primary standard-LCS	50	0.8 mL	Syringe Injection	4.0	10 / 20 / 50

Table A-4

Calibration Standard Levels

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.50	25 mL of 5.0 ppbV sec. standard
2	1.0	50 mL of 5.0 ppbV sec. standard
3	5.0	250 mL of 5.0 ppbV sec. standard
4	10	25 mL of 100 ppbV sec. standard
5	20	50 mL of 100 ppbV sec. standard
6	50	125 mL of 100 ppbV sec. standard
7	80	200 mL of 100 ppbV sec. standard
8	100	250 mL of 100 ppbV sec. standard

Table A-6

ENTECH 7016CA/7100A Operating Parameters for CTD Method

Module 1 (Blank Trap)	
Parameter	**Setting**
Trapping Temperature	-20° C
Internal standard volume	100 mL
Internal standard flow rate	60 mL / min
Sample volume (may vary depending on sample concentrations)	250 mL
Sample flow rate	100 mL / min
Preheat Temperature	10° C
Desorb Temperature	10° C
Bake Temperature	220° C
Bake Time	10 min.
Module 2 (Tenax trap)	
Parameter	**Setting**
Trapping Temperature	-70° C
Desorb Temperature	180° C
Bake Temperature	220° C
Module 2 to Module 3 desorb time	3.5 min
Module 3 (Cryofocusser)	
Parameter	**Setting**
Cryofocusing Temperature	-130° C
Desorb Temperature	Approx. 70° C
Module 3 to GC desorb time	2 min
Bake temperature / event	Approx. 90° C / event 3
Delay time	10 min

Table A-7

Internal Standard (IS) Assignments and Quantitation Ions for Sulfides & Mercaptans

Compound	Quant Ion	Sec. Ion(s)
Bromochloromethane (IS)	49	130
Hydrogen sulfide	34	33, 36
Carbonyl sulfide	60	62, 32
Methyl mercaptan	47	48, 45
ethyl mercaptan	62	47, 29
dimethyl sulfide	62	45, 47
carbon disulfide	76	44, 78
Isopropyl Mercaptan	43	41, 76
tert-Butyl Mercaptan	41	57, 90
n-Propyl Mercaptan	76	47, 43
Ethyl Methyl Sulfide	61	76, 48

Compound	Quant Ion	Sec. Ion(s)
1,4-difluorobenzene (IS)	114	63
Thiophene	84	58, 45
Isobutyl Mercaptan	41	56, 90
diethyl sulfide	75	90, 47
Butyl Mercaptan	56	41, 90
dimethyl disulfide	94	79, 45
1,2-Dichloroethane-D4	65	102

Compound	Quant Ion	Sec. Ion(s)
Chlorobenzene-D5 (IS)	54	82, 117
3-Methylthiophene	97	98, 45
Tetrahydrothiophene	60	88, 45
2-Ethylthiophene	97	112, 45
2,5-Dimethylthiophene	111	112,97
Diethyl Disulfide	122	66, 94
Toluene-D8	98	100
Bromofluorobenzene	95	174

Appendix B

Data Acquistion Parameters and Analysis Modifications for Conducting SIM Analysis

SIM analysis is conducted when full scan sensitivity does not meet the data quality objectives (DQO) of the project and/or regulatory criteria. The acquisition method used to acquire full scan data simultaneously acquires SIM data using the SIM ions and windows specified in Table B-1. The following modifications to the full scan SOP must be done to generate data using the SIM signal:

- SIM level calibration standards must be analyzed w/ the full scan curve (0.02 and 0.04 ppbV)
- A calibration curve is generated using the SIM signal utilizing the lower level calibration standards and must meet the same criteria as the full scan calibration criteria.
- The continuing calibration and/or LCS should be analyzed at a lower concentration (5.0 ppbV)
- Laboratory Method Blanks must be evaluated for the SIM reporting limit as listed in Table B-2.

The SIM signal only acquires data for a limited target analyte list. These target analytes and reporting limits are listed in Table B-2. Additional ions have been added to allow for more analytes to be added, if requested by client. All requirements stated in this SOP must be applied to any TO15-SIM analysis conducted in the laboratory.

Table B-1

Calibration Standard Levels for SIM Analysis

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.02	50 mL of 0.10 ppbV sec. standard
2	0.04	100 mL of 0.04 ppbV sec. standard
3	0.10	250 mL of 0.1 ppbV sec. standard
4	0.20	50 mL of 1.0 ppbV sec. standard
5	0.50	125 mL of 1.0 ppbV sec. standard
6	1.0	250 mL of 1.0 ppbV sec. standard
7	5.0	125 mL of 10 ppbV sec. standard
8	10	250 mL of 10 ppbV sec. standard
9	20	50 mL of 100 ppbV sec. standard
10	50	125 mL of 100 ppbV sec. standard

Table B-2

Seletive Ion Monitoring (SIM) Groupings

Group #	1	2	3	5	6	7	8	9	10	11	12
Group Start Time, min	0	5.9	8.75	10.1	10.9	11.6	13.8	14.6	16.6	18.8	20.5
Dwell time, sec	25	10	25	25	25	15	25	15	3	5	25
Cycles/sec (calc)	2	2.5	3.3	2.1	2.3	1.8	5.7	2.2	7	7	2.3
Analyte Range, Compund #	C2-C7	C8-C20	C21-C26	C27-C29, BCM (IS)	C30-C34	C35-C44, 1,4-DFB (IS)	C45-C46	C47-C55	C56-C67, CHB-D5 (IS)	C68-C76	C77-C80
Ions	29	29 63	43	41	42	39 85	39	39 92	39 106	39	43
	31	31 64	57	43	49	41 87	43	41 94	43 110	43	57
	32	39 66	61	45	51	43 88	58	43 97	45 112	57	63
	39	40 67	63	47	57	45 93	75	45 98	54 114	71	89
	41	41 69	65	49	59	52 95	77	49 100	57 117	75	102
	42	43 72	72	57	62	56 97	100	56 107	75 120	89	109
	43	44 76	73	59	63	57 99		57 109	77 126	91	115
	44	45 84	86	61	64	58 100		58 114	78 128	105	118
	50	47 85	96	70	65	62 110		61 127	82 131	111	128
	51	49 94	98	77	67	63 114		73 129	83 133	115	134
	52	51 96	117	83	71	69 117		75 131	85 156	116	141
	54	52 101	198	85	72	73 119		76 166	91 171	117	142
	58	53 103		86	87	75 129		77	95 173	118	145
	62	55 106		87	97	78 130		83	97 174	119	180
	64	56 108		96	102	82 132		85	103 175	120	225
	67	57 151		97	119	83 174		91	104	126	260
	85	58		98		84			105	134	
	87	59		130						142	
	135	61								146	
										156	
										157	

Table B-3

TO15-SIM Target Analytes and Reporting Limits

COMPOUND	CAS #	SIM Reporting Limit, ppbV	SIM Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.020	0.109
1,1,2,2-tetrachloroethane	79-34-5	0.020	0.137
1,1,2-trichloroethane	79-00-5	0.020	0.109
1,1-dichloroethane	75-34-3	0.020	0.081
1,1-dichloroethene	75-35-5	0.020	0.079
1,2,4-trimethylbenzene	95-63-6	0.020	0.098
1,2-dibromoethane	106-93-4	0.020	0.154
1,2-dichlorobenzene	95-50-1	0.020	0.120
1,2-dichloroethane	107-06-2	0.020	0.081
1,2-dichloropropane	78-87-5	0.020	0.092
1,3,5-trimethylbenzene	108-67-8	0.020	0.098
1,3-butadiene	106-99-0	0.020	0.044
1,4-dichlorobenzene	106-46-7	0.020	0.120
1,4-dioxane	123-91-1	0.10	0.360
benzene	71-43-2	0.10	0.223
bromodichloromethane	75-27-4	0.020	0.134
bromoform	75-25-2	0.020	0.207
bromomethane	74-83-9	0.020	0.078
carbon tetrachloride	56-23-5	0.020	0.126
chlorobenzene	108-90-7	0.020	0.092
chloroethane	75-00-3	0.020	0.053
chloroform	67-66-3	0.020	0.098
chloromethane	74-87-3	0.200	0.41
cis-1,2-dichloroethene	156-59-2	0.020	0.079
cis-1,3-dichloropropene	10061-01-5	0.020	0.091
dibromochloromethane	124-48-1	0.020	0.170
dichlorodifluoromethane	75-71-8	0.200	0.990
ethylbenzene	100-41-4	0.020	0.087
Freon-113	76-13-1	0.050	0.383

COMPOUND	CAS #	SIM Reporting Limit, ppbV	SIM Reporting Limit, ug/m^3
Freon-114	76-14-2	0.050	0.349
hexachlorobutadiene	87-68-3	0.050	0533
methylene chloride	75-09-2	0.5	1.74
MTBE	1634-04-4	0.020	0.072
m+p-xylene	108-38-3 106-42-3	0.040	0.174
o-xylene	95-47-6	0.020	0.087
styrene	100-42-5	0.020	0.085
tetrachloroethene	127-18-4	0.020	0.136
toluene	108-88-3	0.050	0.188
trans-1,2-dichloroethene	156-60-5	0.020	0.079
trans-1,3-dichloropropene	10061-02-6	0.020	0.091
trichloroethene	79-01-6	0.020	0.107
1,2,4-trichlorobenzene	120-82-1	0.050	0.371
vinyl chloride	75-01-4	0.020	0.051
CT RSR Additional Analytes			
1,1,1,2-tetrachloroethane	630-20-6	0.020	0.137
acrylonitrile	107-13-1	0.500	1.08
isopropyltoluene	99-87-6	0.200	1.10
n-butylbenzene	104-51-8	0.200	1.10
sec-butylbenzene	135-98-8	0.200	1.10
isopropylbenzene	98-82-8	0.200	0.98
2-butanone (MEK)	78-93-3	0.500	1.48
Acetone	67-64-1	1.000	2.35
2-methyl-2-pentanone (MIBK)	108-10-1	0.500	2.05
All reporting limits are subject to change.			
Project Specific Analytes			
Naphthalene	91-20-3	0.050	0.262
Halothane	151-67-7	0.050	0.403
1,2,3-trichlorobenzene	87-61-6	0.050	0.371

The following analytes are also reportable via SIM, but at the same reporting limit as full scan:

Propylene, ethanol, isopropyl alcohol, carbon disulfide, 3-chloroprene, vinyl bromide, vinyl acetate, hexane, cylcohexane, tetrahydrofuran, ethyl acetate, 2,2,4-trimethylpentane, and 2-hexanone.

Appendix C

Modifications to Data Review and Case Narrative to Comply with MADEP MCP TO-15 Method

This addendum addresses modifications to this SOP required to be in compliance with the MADEP MCP TO-15 method, specifically "Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Organic Compounds in Air Samples (TO-15) by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)" Revision No. 0, Section IX B.

1) For duplicate analyses, the 25% RPD criteria stated in Sect. 9.6 of this SOP does not need to be applied to concentrations less than 5X the reporting limit.

2) Samples cannot be analyzed if any of the target analytes if the LCS recovery criteria stated in sect. 9.2 of this SOP is below 70% recovery. For compounds listed as difficult analytes (hexachlorobutadiene, 1,2,4-trichlorobenzene, naphthalene, acetone, and 1,4-dioxane), LCS recovery cannot be less than 50%.

3) Any analyte exceeding %RSD criteria of 30% during initial calibration that is a not listed in the MCP TO-15 method, but may still be reported to the client, must be noted in the case narrative.

Appendix D

Method Modifications to Perform the NJDEP TO-15 Low Level Method

The NJDEP-SRP Low Level USEAP TO-15 Method (NJDEP-LL TO-15-3/2007) requires modifications to data acquisition, data processing, and quality control requirements to be compliant with the method.

- **Data Acquisition**
  - scan range has been changed to 35-300 amu and no SIM signal is acquired.
  - The acquisition method is identified as TO15-NJ.M in the Chemstation software.
  - Calibration levels need to be modified from the current SOP. Maximum concentration of the linear range is 40 ppbV (80 ppbV for m+p-Xylene). See table D-1A and D-1B for preparation of secondary standards and required calibration levels.
- **Data Processing**
  - The quantitation ion for ethanol must be changed to 45 due to the change in scan range (normal quantitation ion is 31).
  - Methyl Methacrylate is a target analyte, and the quantitation ion for this compound is 41, with 69 and 100 utilized as secondary ions. It is associated with the internal standard 1,4-Difluorobenzene.
  - Tentatively Identified Compounds (TICs) – a maximum of 30 non-alkane and non-alkene TICs is to be reported for Laboratory Method Blanks, canister certifications, and samples. However, a summation of the concentration of all the alkenes and alkanes detected must be reported.
- **Quality Control Requirements**
  - BFB Tune Check –same requirements as stated in this SOP, both ion abundances and frequency.
  - Initial calibration acceptance criteria-minimum 5 point calibration. %RSD for response factors must be less than 30%, with only two analytes allowed to exceed to 40% RSD.
  - Initial Calibration Verification (ICV) - average response factor must be within +/- 30% of initial calibration (allowance for 2 compounds up to ≤ 40%).and must be a second-source standard or standard prepared from a different lot. If any exceedences occur, repeat the ICV analysis. If second ICV is not within criteria, investigate possible causes for failure and/or re-calibrate.
  - Continuing Calibration Verification (CCV)- average response factor must be within +/- 30% of initial calibration. If any exceedences occur, repeat the CCV

analysis. If second CCV is not within criteria, investigate possible causes for failure and/or re-calibrate.

- A daily LCS check is not required, however a Reporting Limit Laboratory Control Sample (RLLCS) is required. The required concentration to be analyzed is in Table D-2. Acceptance criteria: the percent recovery must be within 60-140% of the known value for 90% of the compounds.

- Although a sample duplicate analysis is not specified in the NJ TO-LL Method, the NJDEP 2014 Technical Guidance, **"NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014**" specifies a duplicate analysis is now required. The duplicate analysis must be performed on a site field sample. RPDs must be ≤ 25% for results > 5x the reporting limit. Duplicate analysis must be performed every 24 hours.

- A closing calibration check must be analyzed within the 24 hour tune window established by the BFB injection time. If any exceedances occur, repeat the closing CCV analysis; this second injection must also be within the 24 hour tune window. If the second closing CCV fails acceptance criteria, laboratory must note exceedance(s) in the case narrative of the report.

Table D-1A

Calibration Standard Levels for NJ-TO15 Analysis

Primary Standard	Primary Standard Concentration	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume Canister (L)	Final Concentration ppbV
TO-15 Mix #1, TO-15 Mix #2, and TO-15 NJ Custom Mix *	1.0 ppmV	1200 mL	50 mL/min for 24 min	12	100
TO-15 Mix #1, TO-15 Mix #2, and TO-15 NJ Custom Mix	1.0 ppmV	120 mL	50 mL/min for 2.4 min	12	10.0
100 ppbV secondary standard	100 ppbV	60 mL	Syringe Injection	12	0.50

* The TO-15 NJ mix is a NIST-tracable standard purchased from Linde. It contains butane, methyl methacrylate, ethanol, tert-butyl alcohol, and 2-chlorotoluene at 1.0 ppmV. Refer to sect. 8.8 & 8.9 of the SOP for detailed instructions of preparation of secondary standards.

Table D-1B

Calibration Standard Levels for NJ-TO15 Analysis

Calibration Level	Amount (ppbV)	Volume / Secondary Standard *
1	0.2	100 mL of 0.5 ppbV sec. standard
2	0.5	250 mL of 0.5 ppbV sec. standard
3	1.0	25 mL of 10.0 ppbV sec. standard
4	5.0	125 mL of 10.0 ppbV sec. standard
5	10.0	250 mL of 10.0 ppbV sec. standard
6	20.0	125 mL of 40.0 ppbV sec. standard
7	40.0	250 mL of 40.0 ppbV sec. standard

Table D-2

True Values for RLLCS Analyses

Analyte	True Value (ppbV)
m+p-xylene	0.40
isopropanol	0.50
tert-butyl alcohol	0.50
methylene chloride	0.50
methyl ethyl ketone	0.50
Tetrahydrofuran	0.50
1,4-dioxane	0.50
methyl methacrylate	0.50
methyl isobutyl ketone	0.50
ethanol	1.0
acetone	1.0
1,2,4-trichlorobenzene	0.50
All other analytes listed in Table D-3	0.20

Table D-3

NJ-TO-15 Target Analytes and Reporting Limits

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.20	1.09
1,1,2,2-tetrachloroethane	79-34-5	0.20	1.37
1,1,2-trichloroethane	79-00-5	0.20	1.09
1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)	76-13-1	0.20	1.53
1,1-dichloroethane	75-34-3	0.20	0.81
1,1-dichloroethene	75-35-5	0.20	0.79
1,2,4-trichlorobenzene	120-82-1	0.20	1.48
1,2,4-trimethylbenzene	95-63-6	0.20	0.98
1,2-dibromoethane	106-93-4	0.20	1.54
1,2-dichlorobenzene	95-50-1	0.20	1.20
1,2-dichloroethane	107-06-2	0.20	0.81
1,2-dichloropropane	78-87-5	0.20	0.92
1,3,5-trimethylbenzene	108-67-8	0.20	0.98
1,3-butadiene	106-99-0	0.20	0.44
1,3-dichlorobenzene	541-73-1	0.20	1.20
1,4-dichlorobenzene	106-46-7	0.20	1.20
1,4-dioxane	123-91-1	0.50	1.80
2,2,4-trimethylpentane	540-84-1	0.20	0.93
2-chlorotoluene	95-49-8	0.20	1.03
4-ethyltoluene	622-96-8	0.20	0.98
acetone	67-64-1	1.0	2.34
allyl chloride (3-chloropropene)	107-05-1	0.20	0.63
butane	106-97-8	0.20	0.48
benzene	71-43-2	0.20	0.64
bromodichloromethane	75-27-4	0.20	1.34
bromoform	75-25-2	0.20	2.07
bromomethane	74-83-9	0.20	0.78
carbon disulfide	75-15-0	0.20	0.62
carbon tetrachloride	56-23-5	0.20	1.26
chlorobenzene	108-90-7	0.20	0.92
chloroethane	75-00-3	0.20	0.53
chloroform	67-66-3	0.20	0.98

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
chloromethane	74-87-3	0.20	0.41
cis-1,2-dichloroethene	156-59-2	0.20	0.79
cis-1,3-dichloropropene	10061-01-5	0.20	0.91
cyclohexane	110-82-7	0.20	0.69
dibromochloromethane	124-48-1	0.20	1.7
dichlorodifluoromethane	75-71-8	0.20	0.99
1,2-Dichlorotetrafluoroethane (Freon-114)	76-14-2	0.20	1.4
ethanol	64-17-5	2.5	4.70
ethylbenzene	100-41-4	0.20	0.87
hexachlorobutadiene	87-68-3	0.20	2.13
hexane	110-54-3	0.20	0.7
isopropanol	67-63-0	0.50	1.23
methylene chloride	75-09-2	0.50	1.74
methyl ethyl ketone (2-butanone)	78-93-3	0.50	1.47
methyl isobutyl ketone	108-10-1	0.50	2.05
Methyl methacrylate	80-62-6	0.50	2.05
methyl tert-butyl ether	1634-04-4	0.20	0.72
m+p-xylene	108-38-3 106-42-3	0.40	1.74
n-heptane	142-82-5	0.20	0.82
o-xylene	95-47-6	0.20	0.87
styrene	100-42-5	0.20	0.85
tert-butyl alcohol	75-65-0	0.50	1.52
tetrachloroethene	127-18-4	0.20	1.36
tetrahydrofuran	109-99-9	0.50	1.47
toluene	108-88-3	0.20	0.75
trans-1,2-dichloroethene	156-60-5	0.20	0.79
trans-1,3-dichloropropene	10061-02-6	0.20	0.79
trichloroethene	79-01-6	0.20	0.91
trichlorofluoromethane	75-69-4	0.20	1.07
vinyl bromide	593-60-2	0.20	0.70
vinyl chloride	75-01-4	0.20	0.87
All reporting limits are subject to change.			

Appendix E

Modifications to Data Review and Case Narrative to Comply with 2014 NJDEP Technical Guidance for EPA Method TO-15

This addendum addresses modifications to this SOP required to be in compliance with the NJDEP 2014 Technical Guidance, **"NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014**". This guidance is to be used when analyzing samples via the EPA Method TO-15.

1) Per the Data of Know Quality Protocols Technical Guidance, surrogates are not required to be reported via EPA Method TO-15.

2) A target analyte list and reporting limits are specified in Table E-1, per the Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance.

3) For the analytes ethanol and isopropyl alcohol, results are allowed to be reported outside the calibration range of the instrument, per the Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance. Results reported that exceed the calibration range will be designated with an "E" qualifier.

4) **Laboratory Control Sample (LCS) criteria -** Must contain all target analytes, recovery range is 70-130%. Exceptions for difficult analytes (hexachlorobutadiene, 1,2,4-trichlorobenzene, naphthalene, acetone, dichlorodifluoromethane, and 1,4-dioxane) must exhibit percent recoveries between 40-160%. In addition, the CCAL analysis cannot be reported as the LCS even if it meets LCS requirements. The LCS must be a separate analysis, analyzed after a passing CCAL standard.

5) **Duplicate analyses** - The 25% RPD criteria stated in Sect. 9.6 of this SOP does not need to be applied to concentrations less than 5X the reporting limit.

6) **Tentatively Identified Compounds (TICs)** – up to 15 TICs must be reported, if present. If a reduced target analyte list is requested by client, TICs are not reported.

Table E-1

NJDEP 2014 Target Analytes and Reporting Limits via EPA Method TO-15

Required Compound Name	CAS Number	Molecular Weight	Reporting Limit ppbV	Reporting Limit ug/m^3
Acetone	67-64-1	58.08	5.0	12
Allyl chloride	107-05-1	76.53	0.2	0.6
Benzene	71-43-2	78.11	0.2	0.6
Bromodichloromethane	75-27-4	163.8	0.2	1
Bromoform	75-25-2	252.8	0.2	2
Bromomethane	74-83-9	94.94	0.2	0.8
1,3-Butadiene	106-99-0	54.09	0.2	0.4
Chlorobenzene	108-90-7	112.6	0.2	0.9
Chloroethane	75-00-3	64.52	0.5	1
Chloroform	67-66-3	119.4	0.2	1
Chloromethane	74-87-3	50.49	0.5	1
Carbon disulfide	75-15-0	76.14	0.5	2
Carbon tetrachloride	56-23-5	153.8	0.2	1
2-Chlorotoluene	95-49-8	126.6	0.2	1
Cyclohexane	110-82-7	84.16	0.2	0.7
Dibromochloromethane	124-48-1	208.3	0.2	2
1,2-Dibromoethane	106-93-4	187.9	0.2	2
1,2-Dichlorobenzene	95-50-1	147.0	0.2	1
1,3-Dichlorobenzene	541-73-1	147.0	0.2	1
1,4-Dichlorobenzene	106-46-7	147.0	0.2	1
Dichlorodifluoromethane	75-71-8	120.9	0.5	2
1,1-Dichloroethane	75-34-3	98.96	0.2	0.8
1,2-Dichloroethane	107-06-2	98.96	0.2	0.8
1,1-Dichloroethene	75-35-4	96.94	0.2	0.8
1,2-Dichloroethene (cis)	156-59-2	96.94	0.2	0.8
1,2-Dichloroethene (trans)	156-60-5	96.94	0.2	0.8
1,2-Dichloropropane	78-87-5	113.0	0.2	0.9
1,3-Dichloropropene (cis)	10061-01-5	111.0	0.2	0.9
1,3-Dichloropropene (trans)	10061-02-6	111.0	0.2	0.9
1,2-Dichlorotetrafluoroethane	76-14-2	170.9	0.2	1
1,4-Dioxane	123-91-1	88.12	5	18
Ethanol	64-17-5	46.07	5	9
Ethylbenzene	100-41-4	106.2	0.2	0.9
4-Ethyltoluene	622-96-8	120.2	0.2	1
n-Heptane	142-82-5	100.2	0.2	0.8
1,3-Hexachlorobutadiene	87-68-3	260.8	0.2	2
n-Hexane	110-54-3	86.17	0.2	0.7
Isopropanol	67-63-0	60.10	5	12

Table E-1 continued

Required Compound Name	CAS Number	Molecular Weight	Reporting Limit ppbV	Reporting Limit ug/m^3
Methylene chloride	75-09-2	84.94	0.5	2
Methyl ethyl ketone	78-93-3	72.11	0.5	1
Methyl isobutyl ketone	108-10-1	100.2	0.5	2
Methyl methacrylate	80-62-6	100.1	0.5	2
Methyl tert-butyl ether	1634-04-4	88.15	0.2	0.7
Styrene	100-42-5	104.1	0.2	0.9
Tert-butyl alcohol	75-65-0	74.12	5	15
1,1,2,2-Tetrachloroethane	79-34-5	167.9	0.2	1
Tetrachloroethene	127-18-4	165.8	0.2	1
Tetrahydrofuran	109-99-9	72.11	5	15
Toluene	108-88-3	92.14	0.2	0.8
1,2,4-Trichlorobenzene	120-82-1	181.5	0.5	4
1,1,1-Trichloroethane	71-55-6	133.4	0.2	1
1,1,2-Trichloroethane	79-00-5	133.4	0.2	1
Trichloroethene	79-01-6	131.4	0.2	1
Trichlorofluoromethane	75-69-4	137.4	0.2	1
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	187.4	0.2	2
1,2,4-Trimethylbenzene	95-63-6	120.2	0.2	1
1,3,5-Trimethylbenzene	108-67-8	120.2	0.2	1
2,2,4-Trimethylpentane	540-84-1	114.2	0.2	0.9
Vinyl bromide	593-60-2	106.9	0.2	0.9
Vinyl chloride	75-01-4	62.50	0.2	0.5
Xylenes (m&p)	179601-23-1	106.2	0.5	2
Xylenes (o)	95-47-6	106.2	0.2	0.9
Naphthalene (reported on request)	91-20-3	128.2	0.2	1

All reporting limits are subject to change.

Appendix F

Modifications to EPA TO-15 SOP to comply with Ohio EPA Voluntary Action Program(VAP) Requirements

This Appendix defines requirements that are necessary to perform TO-15 analysis for any samples submitted via the VAP Program. No deviations from the SOP are allowed under the VAP program.

1) Sec. 3 caveat – reporting limits defined in this SOP are subject to change.

2) Sec. 7 caveat – Equipment and supplies are subject to change.

3) Sec. 2.1 initial calibration acceptance – %RSD criteria must be as defined in the EPA TO-15 method; only 2 analytes allowed greater than 30% RSD, and must be less than 40%.

4) The Laboratory check standard (LCS) may not be used as the continuing calibration verification (CCV). A CCV and LCS must be analyzed prior to samples.

5) Tentatively Identified Compounds (TICs) may not be reported as certified values.

6) CCV-10% rule not allowed. All analytes reported must be +/- 30% D.

7) 10% Duplicate criteria not allowed.All duplicate %RPD results must be below 25%.

8) For Ohio projects, the laboratory standard TO-15 list will be reported, unless otherwise specified by the client.

9) Affidavits are required with each report or for a series of reports generated for a particular project.