

U.S. Army Corps of Engineers Alaska District



NORTHEAST CAPE HTRW REMEDIAL ACTIONS Northeast Cape, St. Lawrence Island, Alaska Contract No. W911KB-10-C-0002 FUDS No. F10AK096903_07.04_0503_p

SAMPLING AND ANALYSIS PLAN REVISION 1 JULY 2010

Submitted by:



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ACRONYMS AND ABBREVIATIONS

%R	percent recovery
'	minutes
0	degrees
A2LA	American Association for Laboratory Accreditation
ADEC	Alaska Department of Environmental Conservation
AK	State of Alaska Method
Bristol	Bristol Environmental Remediation Services, LLC
BTEX	benzene, toluene, ethylbenzene, and xylenes
CCV	continuing calibration verification
CDFR	Chemical Data Final Report
CFR	Code of Federal Regulations
CoC	chain-of-custody
COELT EDF	Corps of Engineers Loading Tool Electronic Data File Format
DO	dissolved oxygen
DoD	U.S. Department of Defense
DoD QSM	U.S. Department of Defense Quality Systems Manager
DOT	U.S. Department of Transportation
DQO	data quality objective
DRO	diesel range organics
DUs	decision units
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EM	Engineer Manual
EPA	U.S. Environmental Protection Agency
ER	Engineering Requirement
FSP	Field Sampling Plan
FVD	fluorescence vs. depth
GC	gas chromatography
GRO	gasoline range organics

ACRONYMS AND ABBREVIATIONS (continued)

HTRW	hazardous, toxic, and radioactive waste				
IS	internal standard				
LIF	laser-induced fluorescence				
LOD	limit of detection				
LOQ	limit of quantitation				
mg/kg	milligrams per kilogram				
MNA	monitored natural attenuation				
MOC	Main Operations Complex				
MS	matrix spike				
MSD	matrix spike duplicate				
NE Cape	Northeast Cape				
NIST	National Institute of Standards and Technology				
NPDL	North Pacific Division Laboratory				
NTUs	nephelometric turbidity units				
ORP	oxygen reduction potential				
PAHs	polynuclear aromatic hydrocarbons				
PCBs	polychlorinated biphenyls				
PE	performance evaluation				
POL	petroleum, oil, and lubricants				
QA	quality assurance				
QAPP	Quality Assurance Project Plan				
QAR	Quality Assurance Representative				
QC	quality control				
RCRA	Resource Conservation and Recovery Act				
RF	response factors				
RIs	remedial investigations				
RPD	relative percent difference				
RRO	residual range organics				
SAP	Sampling and Analysis Plan				

ACRONYMS AND ABBREVIATIONS (continued)

SEDD	Staged Electronic Data Deliverable
SOPs	Standard Operating Procedures
SW	EPA Solid Waste Method
TestAmerica	TestAmerica Laboratories, Inc.
TOC	total organic carbon
TSCA	Toxic Substance Control Act
USACE	U.S. Army Corps of Engineers, Alaska District
UVOST [™]	Ultraviolet Optical Screening Tool
WMI	Waste Management, Inc.

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1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been developed for approval by the U.S. Army Corps of Engineers (USACE), Alaska District, as a control mechanism for the work to be performed on Contract No. W911KB-10-C-0002 for the Hazardous, Toxic, and Radioactive Waste (HTRW) Remedial Actions at Northeast Cape (NE Cape), St. Lawrence Island, Alaska (Figures 1 and 2). The USACE has contracted with Bristol Environmental Remediation Services, LLC (Bristol), and its team of subcontractors, to accomplish the proposed remediation actions. The SAP contains a Field Sampling Plan (FSP) that presents technical details and direction for sampling and field measurement activities, and a Quality Assurance Project Plan (QAPP) that provides quality control (QC) for sampling and analytical activities.

The work includes the following:

- Preparing plans and reports;
- Mobilizing/demobilizing to/from the NE Cape site;
- Excavation, processing and disposal of petroleum-contaminated soils at Site 1 Airstrip, Site 3 Fuel Pump house, Site 6 Former Drum Field, and Site 32 Lower Tramway;
- Excavation, processing, and disposal of polychlorinated biphenyls (PCB)contaminated soils at Site 13 Power Plant, Site 16 Paint and Dope Storage Building, Site 21 Wastewater Treatment Plant, and Site 31 White Alice Communications Station;
- Excavation, processing, and disposal of arsenic-contaminated soil at Site 21 Wastewater Treatment Tank;
- Cleaning and removing a manhole and plugging a culvert located at the western and middle drainages of Site 28 to prevent direct outflows of upgradient residual sources of contamination;
- Sampling tundra/sediment locations for petroleum hydrocarbons at Site 3, following of Alaska Department of Environmental Conservation (ADEC) Technical Memorandum (Tech Memo) 06-001 rationale: Biogenic Interference and Silica Gel Cleanup (ADEC, 2006) and excavating, if necessary, as an optional task;
- Monitoring Natural Attenuation parameters at Site 8 petroleum, oil, and lubricants (POL) Spill Site and collecting 2 surface water samples in the stream flowing from Site 8 into the Suqitughneq River;
- Utilization of Ultraviolet Optical Screening Tool (UVOST) technology to better characterize and delineate subsurface diesel range organics (DRO) contamination at the Main Operations Complex (MOC) (Sites 10, 11, 13, 15, 17, 19, 20, and 27);

- Capping of the Site 9 Housing and Operations Landfill and collecting 4 surface water samples downstream of the landfill from the Site 9 stream and in the Suqitughneq River upstream of the confluence with the Site 9 stream before, during, and after the cap is complete;
- Sampling 10 monitoring wells at the MOC;
- Removal of dangerous poles, wires, and other miscellaneous debris from tundra areas site-wide where clearly identified; and
- Removal of submerged debris from streams in the vicinity of Site 9 Housing and Operations Landfill and Site 29 Suqitughneq (Suqi) River.
- Revegetating or stabilizing, as detailed in the approved Storm Water Pollution Prevention Plan, disturbed site areas prior to demobilization or within a timely manner; and
- Preparing a Remedial Action Report, which includes survey and as-built drawings of Site 9, and discussion of all remedial action work to include soil excavation and removal, sediment removal, waste disposal documentation, sample results, debris removal, UVOST investigation, and other relevant project details.

Figure 3 shows the work areas for the project. Additional planning documents developed for work to be performed for the NE Cape project include the following:

- Work Plan, which includes;
 - Waste Management Plan
 - Site Safety and Health Plan
 - Contractor Quality Control Plan
 - Spill Prevention Control and Countermeasures Plan
- Storm Water Pollution Prevention Plan

1.1 SITE HISTORY

The NE Cape site is located on St. Lawrence Island, in the Bering Sea, near the territorial waters of Russia, approximately 135 air-miles southwest of Nome (Figure 1). The Village of Savoonga is the closest community; located 60 miles northwest of the site. The NE Cape site, at 63 degrees (°) 19 minutes (') north, 168° 58' west, is 9 miles west of the northeastern cape of St. Lawrence Island. The NE Cape site originally encompassed 4,800 acres (7.5 square miles). The site is bounded by Kitnagak Bay to the northeast, Kangighsak Point to the northwest, and the Kinipaghulghat Mountains to the south (Figure 2). The site has been

subject to previous remedial investigations. Due to the remoteness of the St. Lawrence Island site, the field season is generally limited to 90-120 days.

Environmental investigations and cleanup activities at NE Cape began in the mid 1980s with the goal of locating and identifying areas of contamination and gathering enough information to develop a cleanup plan. Remedial investigations (RIs) were initiated at NE Cape during the summer of 1994. Additional sampling was performed during subsequent investigations: Phase II RI (1996 and 1998); Phase III RI (2001 and 2002); and Phase IV RI (2004). The studies divided the concerns among 34 separate sites. The results of the RI showed that contaminants were present at some, but not all sites.

The MOC at the NE Cape installation included the majority of the site infrastructure, including buildings, heat and power supply, fuel storage tanks, maintenance, and housing quarters. Individual MOC sites were grouped together to evaluate an overall response action for the known contamination, which is mostly diesel. These sites are located on the northeast portion of the main complex gravel pad and include Sites 10, 11, 13, 15, 19, and 27. See Figure 3 for work areas.

Bristol performed several removal actions at NE Cape in 2003 and 2005, and in 2009 capped the Site 7 Cargo Beach Landfill and conducted a Phase I In-Situ Chemical Oxidation Study at the MOC.

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2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The project organization is shown in Figure 4. Key positions associated with field sampling and chemical quality assurance (QA) are described below. Resumes for key Bristol personnel are presented in Appendix A. Certificates for key personnel are provided in the Work Plan.

2.1 PROJECT MANAGER

The Bristol Project Manager, Ms. Molly Welker, is the overall manager of technical and administrative activities and is responsible for coordinating and scheduling all project activities, implementing the terms and conditions of this SAP, and interfacing with the USACE and agency personnel. This will include generating, approving, and issuing project-level instructions, procedures, drawings, calculations, specifications, and reports necessary to accomplish assigned tasks.

2.2 SITE SUPERINTENDENT/SITE SAFETY AND HEALTH OFFICER

Mr. Charles (Chuck) Croley is responsible for management of scheduling, coordination, and execution of Bristol on-site activities in accordance with the contract specifications. He will report directly to the Project Manager. Mr. Croley will be Bristol's on-site representative in dealings with subcontractors and deal with all safety issues.

2.3 PROJECT CHEMIST

The Project Chemist, Mr. Marty Hannah, has the responsibility for project-related quality aspects concerning the collection and chemical analysis of all samples, as delegated by the Project Manager. His primary roles are to provide oversight to the data development and review process and all subcontracted laboratories, as well as managing the on-site field-screening laboratory. He will report directly to the Construction Quality Control Site Manager in the field.

Mr. Hannah is responsible for ensuring the project meets the data quality objectives (DQOs) outlined in the QAPP and for developing a detailed scope of work for the subcontract laboratory that incorporates the DQOs as described in Section 5.2 of this SAP. He will direct laboratory audits, as necessary, and data validation activities to ensure the DQOs, as described in this SAP, are satisfied.

2.4 Environmental Samplers

The Environmental Samplers are responsible for the day-to-day sampling activities for their respective operations units in the field. They will coordinate directly with the Site Superintendent and Project Manager to implement all operations aspects of the project planning documents. The lead Environmental Sampler is Mr. Eric Barnhill. Ms. Lyndsey Kleppin, will serve as the Alternate Environmental Sampler.

2.5 SUBCONTRACTORS

2.5.1 General Subcontractors

All subcontractors involved with data generation will be required to comply with the QA requirements of the QAPP, located in Section 5.0.

2.5.2 Analytical Laboratory

The project will have laboratory support from a commercial environmental analytical laboratory, TestAmerica Laboratories, Inc. (TestAmerica), in Tacoma, Washington. Ms. Terri Torres, the Client Service Manager, has been assigned as the program Laboratory QA Officer for this project. She is responsible for enforcement of the Laboratory OA Plan. This includes the internal review of analytical results and verification that all analytical protocols follow the specified U.S. Environmental Protection Agency (EPA) or State of Alaska (AK) methods, using the laboratory-implemented Standard Operating Procedures (SOPs), and meeting the DQOs as specified in this SAP. Ms. Torres is also responsible for ensuring the laboratory meets criteria set forth in the Department of Defense Quality Systems Manual, version 4.1 (DoD QSM 4.1). She will also serve as the Laboratory Project Manager. Ms. Torres has a Bachelor of Science, in Biology from The Evergreen State College in Olympia, Washington, and over 14 years' experience in the analytical services field. Ms. Christina Mott, the Quality Assurance Manager, is responsible for maintaining current USACE laboratory certifications and the Laboratory QA Plan. Ms. Mott has a Bachelor of Arts, in Chemistry and over 18 years' experience in the environmental chemistry field. The Laboratory QA Officer, in conjunction with the Laboratory Director, Ms. Kathy Kreps, will ensure that the laboratory is staffed with qualified personnel and that adequate facilities and instrumentation are available to conduct the work in accordance with detailed requirements found in the USACE Engineer

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Manual (EM) 200-1-3 (2001). TestAmerica's certifications for laboratory work are presented in Appendix B. The TestAmerica laboratory in Tacoma has received its DoD Environmental Laboratory Accreditation Program (ELAP) accreditation, which is required before performing any analyses for this project.

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3.0 FIELD SAMPLING ACTIVITIES

This section details the field sampling activities to be conducted at NE Cape in the work areas shown on Figure 3. Table 3-1 lists the work areas along with their estimated sample area, target parameters, and the approximate tons of soil to be excavated from each area.

Work Area	Site Descriptions	Estimated Sample Area (sq ft)	Contaminant of Concern	Estimated Tons of Soil to be Excavated
Site 1	Airstrip	650	RRO	100
Site 3		750	DRO	100
Site 5	Fuel Pump House	750	RRO	- 100
Site 6	Cargo Beach Road Drum Field	14,000	DRO	2,500
Site 8	POL Spill	12,500	DRO	None
Site 9	Landfill Cap	2.1 acres ¹	Metals DRO/RRO	NA
Site 13	Power and Heat Building	2,500	PCBs	225
Site 16	Paint and Dope Storage Building	300	PCBs	15
Site 21	Wastewater	600	PCBs	15
Site 21	Treatment Tank	600	Arsenic	15
			PCBs	
Site 28	Manhole/Culvert at Drainage Basin	NA	Arsenic	NA
			DRO	1

Table 3-1 WORK Areas	Table	3-1	Work Areas
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Work Area	Site Descriptions	Estimated Sample Area (sq ft)	Contaminant of Concern	Estimated Tons of Soil to be Excavated
Site 31	White Alice Communications Station	900	PCBs	175
Site 32	Lower Tram	750	DRO	25
Main Operations Complex (Site 10, 11, 13, 15, 19, 27)	UVOST [™]	5.9 acres ²	DRO ³	NA

Table 3-1Work Areas (continued)

Notes:

¹Estimated landfill cap area

² Estimated area that will be investigated by the UVOST

³Primary contaminant of concern in soils

	-				
DRO	=	diesel range organics	RRO	=	residual range organics
NA	=	not applicable	sq ft	=	square feet
PCBs	=	polychlorinated biphenyls	UVOST	=	Ultraviolet Optical Screening Tool
POL	=	petroleum, oil, and lubricants			

Table 3-2 identifies the work areas, contaminants of concern and their required analytical methods, cleanup levels, Limit of Quantitation (LOQs), and Limits of Detection (LODs) in the fixed based laboratory for each work area. The cleanup levels were established for the site in the Decision Document for the HTRW Project # F10AK096903, Northeast Cape FUDS, St. Lawrence Island, Alaska, January 2009 (USACE, 2009).

Work Area	Contaminant of Concern	Analytical Test Method	Soil Cleanup Levels (mg/kg)	Sediment Cleanup Levels (mg/kg)	Surface Water Cleanup Levels (mg/L)	LOQs (mg/kg)	LODs (mg/kg)
Site 1	RRO	AK 103	9,200	NA	NA	50	6.17
0.1	DRO	AK102	9,200		NA	20	2.3
Site 3	RRO	AK103	9,200	3,500	NA	50	6.17
Site 6	DRO	AK102	9,200	3,500	NA	20	2.3
Site 8	DRO	AK102	NA	3,500	NA	20	2.3
Site 13	PCBs	EPA 8082	1	NA	NA	0.01	0.003
Site 16	PCBs	EPA 8082	1	NA	NA	0.01	0.003
014.04	PCBs	EPA 8082	1	NA	NA	0.01	0.003
Site 21	Arsenic	EPA 6020	11	NA	NA	0.2	0.004
-	PCBs	EPA 8082	NA	0.7	NA	0.01	0.003
Site 28	Arsenic	EPA 6020	NA	93	NA	0.2	0.004
	DRO	AK102	NA	3,500	no sheen	20	2.3
Site 31	PCBs	EPA 8082	1	NA	NA	0.01	0.003
Site 32	DRO	AK102	9,200	NA	NA	20	2.3

Table	3-2	Site	Cleanup	Levels

AK	=	Alaska Method	mg/kg	=	milligrams per kilogram
DRO	=	diesel range organics	mg/L	=	milligrams per liter
EPA	=	U.S. Environmental Protection Agency	NA	=	not applicable
LODs	=	Limits of detection	PCBs	=	polychlorinated biphenyls
LOQs	=	limits of quantitation	RRO	=	residual range organics

Table 3-3 details the estimated soil sample quantities for field-screening samples and laboratory confirmation samples, and the required analyses (primary samples, plus QC samples, matrix spikes (MSs), and matrix spike duplicates (MSDs) and field duplicates for each work area.

Work Area	Contaminant of Concern	Analytical Test Method		Screening mples	Conf	alytical irmation mples ¹	Field Duplicates	MS/ MSD
			Floor	Sidewall	Floor	Sidewall		
Site 1	RRO	AK103	.8	4	4	1	1	1
	DRO	AK102	16	4	4	2	6	3
	RRO	AK103	16	4	4	2		
Site 3	DRO-SG	AK102- SG	4	0	2	0	1	1
	тос	EPA method 9060	16	4	4	2	6	3
	RRO-SG	AK103-SG	4	0	2	0	1	1
Site 6	DRO	AK102	100	20	46	11	6	3
	DRO	AK102	8**	0	3	0	1	1
	RRO	AK103	8**	0	3	0	1	1
	DRO-SG	AK102-SG	0	0	3	0	1	1
Site 8 ²	RRO-SG	AK103-SG	0	0	3	0	1	1
	тос	EPA 9060	0	0	3	0	1	0
	PAHs	EPA 8270C SIM	0	0	3	0	1	0
Site 13	PCBs	EPA 8082	40	38	27	4	4	2
Site 16	PCBs	EPA 8082	24	8	9	4	2	1
011-04	PCBs	EPA 8082	8	8	2	0	1	1
Site 21	Arsenic	EPA 6020	0	0	3	1	1	1
0.1 0.0	DRO	AK102	0	0	2*	0	1	1
Site 28	PCBs	EPA 8082	0	0	2*	0	1	1

Table 3-3 Soil Sample Collection Summary

Work Area	Contaminant of Concern	Analytical Test Method		Screening mples	Cont	alytical firmation mples ¹	Field Duplicates	MS/ MSD	
			Floor	Sidewall	Floor	Sidewall			
	Metals ³	EPA 6020	0	0	2*	0	1	1	
Site 31	PCBs	EPA 8082	16	4	10	4	2	1	
Site 32	DRO	AK102	8	2	4	4	1	1	

Table 3-3 Soil Sample Collection Summary (continued)

Notes:

¹All PCB samples will be analyzed either individually or according to the compositing schemes discussed in Section 3.1.1. As long as every sample collected at a grid point is analyzed as either an individual sample or as part of a composite sample, there are no other restrictions on how many samples are analyzed per 40 CFR 761.289, Subpart O.

²There is no excavation at this site. Samples will be collected from a wetland.

³Metals include arsenic, barium, cadmium, chromium, lead, mercury, nickel, selenium, silver, and zinc.

*Potential sediment samples collected from the manhole at Site 28.

**Samples will be analyzed to determine decision unit boundaries based on results and chromatographic interpretation.

AK	=	Alaska Method	MSD	=	matrix spike duplicate
CFR	=	Code of Federal Regulations	PAH	=	polynuclear aromatic hydrocarbons
DRO	=	diesel range organics	PCBs	=	polychlorinated biphenyls
EPA	=	U.S. Environmental Protection Agency	RRO	=	residual range organics
MNA	=	monitored natural attenuation	SG	=	Sample extracts will be treated with silica gel cleanup
			SIM	=	selective ion monitoring
MS	=	matrix spike	TOC	=	total organic carbon

Table 3-4 estimates the surface and ground water samples that will be collected at Sites 6, 8 and 9 and at the MOC.

Work Area	Contaminant of Concern	Analytical Test Method	Primary Samples	Field Duplicates	MS/ MSD
	GRO	AK101	1	1	1
	DRO	AK102	1	1	1
Site 6 ¹	RRO	AK103	1	1	1
	BTEX	EPA 8260B	1	1	1
	PAHs	EPA 8270C- SIM	1	1	1

Table 3-4	Surface and Ground Water Sample Collection Summary
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Work Area	Contaminant of Concern	Analytical Test Method	Primary Samples	Field Duplicates	MS/ MSD
	DRO	AK102	2	1	1
	RRO	AK103	2	1	1
Site 8 [#]	PAHs	EPA 8270C SIM	2	1	1
	MNA	See Notes ³	24	3	0
	Methane	RSK 175	24	3	0
	VOCs	EPA 8260B	12*	2*	3*
	Metals ²	EPA 6020A	12*	2*	3*
	GRO	AK101	12*	2*	3*
Site 9	DRO	AK102	12*	2*	3*
	RRO	AK103	12*	2*	3*
	PAHs	8270C-SIM	12*	2*	3*
	PCBs	EPA 8082	12*	2*	3*
	PCBs	EPA 8082	10	1	1
	VOCs	EPA 8260	10	1	1
	GRO	AK101	10	1	1
MOC ⁴	DRO	AK102	10	1	1
	Metals ²	EPA 6020A	10	1	1
	PAHs	EPA 8270C- SIM	10	1	1
	MNA	See Notes ²	10	1	0

Table 3-4 Surface and Ground Water Sample Collection Summary (continued)

Notes:

¹A sediment and surface water sample will be collected near 94NE06SW/SD115 at Site 6 if possible; if not, a soil sample will be collected.

²Metals include arsenic, barium, beryllium, cadmium, chromium, lead, mercury, nickel, selenium, silver, vanadium and zinc.

³MNA parameters to be field analyzed in ground water are: nitrate, sulfate, ferrous iron, alkalinity and dissolved manganese.

⁴Groundwater samples from 10 monitoring wells.

*Four primary samples will be collected before, during, and after the landfill cap is completed.

[#]There is no excavation at this site. Samples will be collected from a wetland.

AK	=	Alaska Method	MS	=	matrix spike
CFR	=	Code of Federal Regulations	MSD	=	matrix spike duplicate
DRO	=	diesel range organics	PAH	=	polynuclear aromatic hydrocarbons
EPA	=	U.S. Environmental Protection Agency	PCBs	=	polychlorinated biphenyls

Table 3-5 details sample quantities and required analyses necessary for waste characterization and disposal.

Parameter Method		the second se	Primary Samples		Field Duplicates	MS/MSD		
D	RO/F	RO	AK102/AK103	52	6		NA	
RC	RCRA Metals EPA 6020		EPA 6020	2	1		NA	
	PCB	S	EPA 8082	8		1	NA	
Notes: AK DRO EPA	=		Nethod Inge organics vironmental Protection Age	NA PCBs ency RCRA Metals	=	Metals include Ars Cadmium (Cd), Ch	vation and Recovery Act enic (As), Barium (Ba), nromium (Cr), Lead (Pb), enium (Se), Silver (Ag), Nicke	
MS MSD	= =	matrix sp matrix sp	pike pike duplicate	RRO	=	residual range org	anics	

Table 3-5 Soil Sample Quantities for Waste Characterization

3.1 MITIGATION PLANS FOR POL- AND PCB-CONTAMINATED SOIL

The Scope of Work for this contract estimates the tonnage of POL-, PCB-, and arseniccontaminated soil (Table 3-1) to be removed from eight sites at NE Cape. Locations at each site to be excavated are shown on Figures 5 thru 15. At the start of the field effort each location to be excavated at a site will be identified and staked by a professional land surveyor using the government furnished survey data and former control points.

If necessary, contaminated soil will be removed from the sites and transported to Site 6 where a rock-screening plant will remove 2-inch plus material. After screening, the soil will be loaded into the bulk bags using a CAT excavator with a thumb attachment, or a front-end loader. The DRO/residual range organics (RRO)-contaminated sites (Sites 1, 3, 6, and 32) will be excavated first, and then the field crew will excavate the PCB-contaminated soil at Sites 13, 16, 21, and 31. A screen plant staged at Site 6 will be used to shake and sieve the POL-contaminated soil to separate the fine-grain particles that are less than 2 inches in diameter (minus 2-inch material) from the coarser rock material.

The screening plant will be located on contaminated soil at Site 6 during all screening of petroleum-contaminated soils. Eventually, the screening plant will have to be moved in order to process the final remaining areas of POL contamination at Site 6. The new screening area will be sampled prior to moving the screening plant into position at Site 6. Following the completion of screening activities, the area will be sampled again to evaluate the impact of the operation to soil contamination concentrations. These analyses will be done by the on-site field laboratory. Any secondary contamination resulting from screening activities will be remediated by excavating the impacted areas and disposing of the soils off-island.

For PCB-contaminated soils, the screening plant will be set up on a concrete pad at the MOC, with secondary containment to control runoff around the screen plant. Similar to petroleum contaminated soils, material greater than 2 inches in diameter will be segregated out into stockpiles. For the PCB soils, at least one rock sample will be obtained from each 10 cubic yards of stockpiled, oversized material for field screening purposes. Rock samples will be obtained from various depths in the pile. At least 2 rock samples will be collected from stockpiles of 50 cubic yards or less, with at least one additional sample collected from each additional 50 cubic yards of the stockpile or portion thereof over the initial 50 cubic yards. These representative samples from the oversized material stockpiles from each of the PCB-contaminated sites (Sites 13,16, 21, and 31) will be sent to Dowl-Alaska Testlab to be crushed and then shipped to TestAmerica-Tacoma for PCB analysis. If the crushed, oversized material is less than 1.0 milligrams per kilogram (mg/kg), it will be used as backfill at the sites. If the crushed, oversized material has PCB concentrations greater than 1.0 mg/kg, it will be containerized for off-site shipment and disposal.

The field crew assigned to the screen plant, in cooperation with the QAR, will determine when truckloads of the PCB-contaminated soil should be placed directly into bulk bags and not screened due to heavily stained rocks, and/or when frozen material is incorporated with the PCB-contaminated soil.

The concrete pad used to screen out the PCB oversized material will be swept clean at the end of the screening process.

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The minus 2-inch material, and all non-rock oversized material (frozen soil, soil clumps, wood, etc.), will be transported and disposed of off site. Field notes and photographs will be used to document the screening process and whether oversized material is clean (e.g., no petroleum staining, no excessive amounts of fine-grained soils are adhering to the rock, and no non-rock material is screened).

The POL-coarser material will be used as backfill in the POL excavation areas in accordance with ADEC Petroleum Hydrocarbon Cleanup for Oversize Material Technical Memorandum, which states:

Rock material greater than two inches does not require remediation or testing, unless it has the potential to hold excessive amounts of contamination or contains visible petroleum product on the surface (surface stain). The EPA Region 10 PCB Coordinator previously approved that under Title 40 Code of Federal Regulations, part 761.61C (40CFR 761.61C), the PCB rock material (plus 2-inch fraction) can be used as backfill based on laboratory results. Bristol received permission to use the screened PCB-contaminated soil as backfill during the 2007 Hoonah Radio Relay Station Remedial Action Project (Contract W911KB-06-D-0007). Also according to 40 CFR 761.267 (b), it is not necessary to sample small or irregularly shaped surfaces.

After the excavation is complete at each location, soil samples collected from the floor and sidewalls of the excavation will be screened for DRO/RRO or PCBs with gas chromatographs set up in an on-site field laboratory. The on-site field laboratory will be set up in a trailer at the remote camp location adjacent to the runway. The field laboratory SOPs and sample correlation results between the field laboratory and the fixed analytical laboratory are in Appendix C.

Field-screening results will be used to direct the excavation of contaminated soil, but will not be used to determine if site cleanup levels have been met. Once the excavation is believed to be complete based on the field screening results, confirmation samples will be collected and submitted to TestAmerica in Tacoma, Washington, an ELAP-accredited laboratory, to confirm that the remaining soil is below site cleanup levels.

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3.1.1 Soil Field Screening and Laboratory Confirmation Samples

Table 3-1 shows the approximate square footage from each work area that will be excavated. Table 3-3 summarizes the number of field-screening and laboratory confirmation samples that will be collected. The field team will establish a 25-foot by 25-foot sampling grid for discrete field-screening and confirmation samples for the POL excavation areas. Discrete samples will be collected from the sidewalls and floors of the excavations. For the PCB-excavation areas, a 5-foot by 5-foot grid will be established to identify sample locations to meet Toxic Substance Control Act (TSCA) sample regulations.

Discrete samples collected from up to 9 grids will be composited for a PCB field-screening sample. Floor samples from the excavation will be composited from contiguous grids and from the same depth level. Sidewall field-screening samples will be a composite of soil samples. The PCB composite sample will be made up of discrete samples collected one every 5 feet of horizontal distance along the sidewall for PCB excavations.

For both the POL and PCB field screening and confirmation samples, the sample will be collected midway up the wall from each grid at the perimeter of the excavated area. The sidewall depth from the excavated areas will vary from 1 foot to possibly greater than 5 feet deep. The field team will attempt to field screen the most POL-contaminated areas based on visual observations, such as staining and odors, and may use PID readings in the DRO/RRO excavations.

If any field-screening samples contain PCBs above 0.8 mg/kg and any samples contain POL above 7,500 mg/kg, additional soil will be removed from the excavation floor and/or sidewalls.

If all field-screening samples from an excavation are less than 0.8 mg/kg PCB or 7,500 mg/kg DRO/RRO, discrete grid-based samples will be collected and sent to the TestAmerica, Tacoma laboratory as a confirmation sample from each work area. The PCB discrete samples will be composited by the analytical laboratory and documented on the chain-of-custody. The field-screening and soil excavation processes will continue until analytical results from TestAmerica indicate all soil in excess of the cleanup criteria has been removed. If any of the confirmation soil samples contain PCBs above the 1.0 mg/kg cleanup level or DRO/RRO

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above 9,200 mg/kg cleanup level, additional soil from that area of the excavation will be removed. Additional field screening will then be performed to confirm that soil contaminated with PCBs or DRO/RRO above the cleanup level is not present, and additional confirmation samples will be collected.

The Quality Assurance Representative (QAR) will be notified after field-screening and confirmation results indicate that the cleanup levels have been achieved for a location. The excavation will not be backfilled until the QAR has reviewed and approved the confirmation and field screening results. Backfilling procedures are discussed in the Work Plan.

Duplicate samples will be collected at a rate of 10 percent for each site and MS/MSD samples will be collected at a rate of 5 percent for each site. The estimated numbers of duplicate QC samples for each site are shown in Table 3-3.

3.1.2 Site 3 and Site 8 Silica Gel Cleanup Samples

Three soil samples collected at Site 3 and Site 8 will be subjected to silica gel cleanup and total organic carbon (TOC) analyses as described in ADEC Technical Memorandum 06-001 (ADEC, 2006a) when they exhibit biogenic characteristics. Three background samples to evaluate biogenic interference will also be collected at Sites 3 and 8. Additional samples may be collected and analyzed from areas outside of the contaminated area that are representative of the soil within the contaminated area (background samples).

The purpose of the silica gel cleanup procedure is to compare the same soil sample extract and reported concentrations before and after the silica gel procedure. Silica gel removes polar compounds, which are not characteristic of non-weathered petroleum compounds. A trained, experienced chemist will evaluate the TOC and AK102/AK103 results before and after silica gel treatment and interpret the subsequent chromatograms for biogenic characteristics.

3.2 MITIGATION PLANS FOR ARSENIC-CONTAMINATED SOIL AT SITE 21

Arsenic was detected above site cleanup levels at Site 21 at an anomalous concentration of 170 mg/kg at a single location (SS167), located in surface soil downgradient of the septic tank outfall during the 1994 investigation. Sample location SS167 will be identified by a professional land surveyor at the beginning of the field effort. After this location is identified,

approximately 15 tons of arsenic-containing soils will be excavated and placed in the U.S. Department of Transportation (DOT)-approved bulk bags for disposal.

The field team will establish a 25-foot by 25-foot sampling grid for the arsenic field screening and confirmation samples that will be sent to the analytical laboratory. Confirmation sampling procedures will follow those used for the POL confirmation samples.

If samples have arsenic levels above the cleanup level of 11 mg/kg, additional soil will be removed until the sample results are below the cleanup level.

3.3 WASTE CHARACTERIZATION OF EXCAVATED SOIL

Excavated soil will be containerized and characterized for proper manifesting and disposal. Refer to the Waste Management Section in the Work Plan for detailed disposal information. All contaminated soil (minus 2-inch fraction) will be contained in DOT-approved bulk bags. The bags are lined, stackable, and capable of holding approximately 8 cubic yards or approximately 15,000 pounds of soil. Bags will be placed inside a loading frame and filled with soil from each of the work areas. Rock trucks will be used to transport the bags from the work areas to Cargo Beach for staging.

Sampling each bulk bag for waste characterization will occur immediately after the soil is placed in the bulk bags at the work area. Grab samples from seven (7) bags will be composited into one (1) waste characterization sample for field screening by the on-site laboratory. A grab sample will be collected from each end of the bag for a total of two (2) grab samples per bag. The on-site field-screening laboratory will be used to characterize the POL- and PCB-contaminated soil for disposal. Characterization by the field-screening laboratory is acceptable to the disposal facilities.

The excavated PCB-contaminated soil will be characterized by the level of PCBs and segregated as greater than 50 mg/kg PCBs and less than 50 mg/kg PCB waste, and transported off site for disposal. A composite soil sample made up of discrete samples collected from a set of 7 bulk bags will be used to characterize the soil. Bristol will take into consideration the field-screening results and not combine PCB-contaminated soil >50 mg/kg with soil containing <50 mg/kg PCB. If the results from the field-screening laboratory indicate that

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PCB levels are below 45 mg/kg, the soil in the bulk bags will be handled and disposed of as non-TSCA waste. If the results are positive for PCB greater than 45 mg/kg, discrete samples from the bulk bags that made up the composite sample will be collected and analyzed at the field-screening laboratory. Any discrete sample above the 45 mg/kg PCB level will be sent to the fixed-based analytical laboratory to confirm the concentration for disposal purposes, as necessary. The waste will then be correctly manifested as either TSCA or non-TSCA waste. Re-sampling discrete grab samples from the bulk bags will be performed either at the staging area, or Cargo Beach, depending on the location of the container at the time.

One field duplicate will be collected for every 10 composite waste samples. Due to the expected high concentrations of DRO contaminants in the waste soil, no QC samples will be collected or submitted for MS/MSD analyses. Table 3-5 lists the estimated number of primary composite samples and QC samples to be collected for containerized waste.

All POL-contaminated soil in the bulk bags will be transported to a disposal facility operated by Waste Management Inc., in Oregon, as described in the Waste Management Section of the Work Plan. Contaminated soil with PCB concentrations less than 50 mg/kg will be disposed of at the Columbia Ridge Landfill in Arlington, Oregon. Soil with PCB concentrations greater than 50 mg/kg will be shipped and disposed of through Chemical Waste Management of the Northwest in Arlington, Oregon, as described in the Waste Management Section of the Work Plan.

3.3.1 Site 28 Sediment Removal and Waste Characterization

An undetermined amount of sediment resides at the base of a manhole on the western drainage at Site 28 (Figure 13). The sediment will be removed and transferred to 55-gallon drums for characterization and disposal. Discrete samples will be collected from each drum and composited for analysis. The composite sample will be analyzed for DRO/RRO, PCBs, and Resource Conservation and Recovery Act (RCRA) metals (e.g., arsenic, barium, cadmium, chromium, lead, mercury, nickel, selenium, silver and zinc). The analytical results will be used for proper disposal of the sediment and will be used for future remediation plans by the USACE for the cleanup of Site 28 drainage.

3.4 SITE 8 MONITORING NATURAL ATTENUATION PARAMETERS AND SURFACE WATER SAMPLES

Previous investigations at Site 8 identified a fuel pipeline breakage on the west side of the intersection of Airport Road and Cargo Beach Road. The breakage area is bordered on the east by the Airport and Cargo Beach Roads, and a wetland area lies to the west of the former pipeline route and roads (Figure 8). A 2004 sampling investigation identified sediments at sample locations 08SD102 and 08SD103 to be wet, brown organic silt with active roots. Sediments at sample location 08SD103, which is roughly 50 feet south of the suspected pipeline break area, were noted as having a weathered diesel odor. Previous investigations had noted that fuel-impacted soil was evident at the foot of the roadway embankment, and disturbed soil in the pipeline corridor may indicate the spill location.

The Bristol field-sampling crew will divide the wetland area into three sample decision units (DUs). The investigation crew will use field screening, and visual and olfactory senses to identify impacted areas.

This 2010 sampling scheme at Site 8 is designed to create a baseline data set for monitored natural attenuation (MNA) parameters that, according to the USACE, will continue to be sampled and monitored for an initial three years at Site 8. Data from each year of monitoring will be compared to determine the rate of natural attenuation. Measureable attenuation parameters are listed in Table 3-6.

Two surface water samples will be collected in the Site 8 stream that flows into the Suqitughneq River. The sample locations near the confluence with the Suqitughneq River will be selected by the QAR and the Bristol field team leader. The parameters to be analyzed are listed in Table 3-4.

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	Parameter	Matrix	Method	Analytical Samples	QC	MS/MSD ¹
	рН	Water	YSI-Field Measured	24	3	0
	Dissolved Oxygen Water YSI-Field Measured		24	3	0	
	Conductivity	Water	YSI-Field Measured	24	3	0
	Oxidation/Reduction Potential	Water	YSI-Field Measured	24	3	0
	Temperature	Water	YSI-Field Measured	24	3	0
Field Measurements	Turbidity	Water	Hach Nephalometer- Field Measured	24	3	0
	Nitrate	Water	Hach Kit-Field Measured	24	3	0
	Sulfate	Water	Hach Kit-Field Measured	24	3	0
	Ferrous Iron	Water	Hach Kit-Field Measured	24	3	0
	Alkalinity	Alkalinity Water Hach Kit-Field Measured		24	3	0
	Dissolved Manganese	Water	Hach Kit-Field Measured	24	3	0
	Methane	Water	RSK 175	24	3	0 ²
Laboratory	Total Organic Carbon	Sediment	EPA method 9060	3	1	1
Analyses	DRO/RRO	Sediment	AK102/AK103	3	1	1
	DRO-Silica Gel	Sediment	AK102-SG	3	1	1
	RRO-Silica Gel	Sediment	AK103-SG	3	1	1
	PAHs	Sediment	8270c-SIM	3	1	1

Site 8 Sample Summary Table 3-6

Notes:

¹MS/MSDs will be collected for every batch (less than or equal to 20 samples) submitted to the laboratory.

²The laboratory has no approved method for spiking methane samples in water.

AK	=	Alaska Method	MS	=	matrix spike
DRO	=	diesel range organics	MSD	=	matrix spike duplicate
EPA	=	U.S. Environmental Protection Agency	RRO	=	residual range organics

The wetland is approximately 40 feet wide (east-west) and 300 feet long in the study area. The upper (northerly) portion will represent the non-impacted (background) Decision Unit (DU), the center section will represent the high fuel impact DU, and the lower portion toward the Suqi River will likely represent a lower (downgradient) fuel impacted area. Previous studies have not provided sufficient information to determine the magnitude of impact the fuel has had on the affected area.

Once the upper boundary of the impact area has been delineated, a sample grid will be developed for each DU. Each grid will be divided into four sections wide by ten sections long for 40 possible sample points and grid squares measuring approximately 10 feet by 10 feet. A random number generator will select eight sample points for each DU grid for sampling. Procedures for surface water sampling are detailed in Section 4.3.1.

After all of the surface water samples have been collected, soil-sediment samples will be collected for DRO/RRO, TOC, and PAHs analysis from each of the eight sample points in each DU. The eight sediment samples from each DU will then be composited as one sample. A field duplicate will be split from one of the composite samples after the soil sediment has been homogenized. Sediment sample collection procedures are described in Section 4.4. The soil sediment samples will be subjected to silica gel cleanup as described in Section 3.1.1 to evaluate the presence and proportion of biogenic materials at the site.

The primary line of evidence for determining if natural attenuation is occurring at a site is the decrease in the contaminants of concern, in this case POL. The geochemical parameters are used as secondary indications that natural attenuation is occurring. The POL and monitored natural attenuation (MNA) data will be evaluated to determine if natural attenuation is occurring at the site and if it is an adequate remedy to meet cleanup goals. If natural attenuation is occurring, dissolved oxygen (DO), nitrate, and sulfate, should have relatively low or no concentrations detected with field parameters. These compounds provide a source of oxygen (electron acceptors) to facilitate beta-oxidation (aerobic) of organic compounds. It will not determine if oxidation is occurring on natural sources or POL. If anaerobic degradation is occurring, the levels of dissolved ferric iron (Fe²⁺), dissolved manganese (Mn²⁺), and dissolved methane will increase. Alkalinity will also increase in the plume or

source area if microbial activity is occurring as alkalinity is most influenced by CO_2 content in the water. A negative oxygen reduction potential (ORP) value also indicates that natural attenuation is occurring in an anaerobic state.

According to the USACE, the results from this initial attenuation study will be used as a baseline and additional sampling will take place over the next two years, and every five years afterwards. Future sampling will repeat the collection of MNA parameters and contaminant levels in order to more accurately determine if trends are occurring. Statistical models, such as Mann-Kendall, are used to evaluate and model natural attenuation parameters.

3.5 GROUNDWATER MONITORING AT THE MOC

Up to 10 groundwater wells will be sampled for petroleum compounds, metals, benzene, toluene, ethylbenzene, and xylenes (BTEX), GRO, DRO, PCBs, PAHs, and MNA parameters. The groundwater results will establish a baseline data set that will be compared in the future for areas outside of the anticipated areas that may be excavated at the MOC. Table 3-7 lists the sample quantities and specific analytes. Section 3.4 of the SAP provides a discussion of MNA and how the parameters are used to evaluate whether natural attenuation is occurring. The MNA parameters that will be measured in the field are shown in Table 3-6. Additional MNA parameters that will be monitored at the well locations with a YSI water quality meter with flow-through cell are pH, conductivity, DO, and ORP. Sampling procedures for groundwater are detailed in Sections 4.3.2 and 4.3.3.

	Parameter	Matrix	Method	Analytical Samples	QC	MS/MSD ¹
	рН	pH Water YSI-Field Measured		10	1	0
	Dissolved Oxygen Water YSI-Fie		YSI-Field Measured	10	1	0
	Conductivity	Water	YSI-Field Measured	10	1	0
	Oxidation/Reduction Potential	Water	YSI-Field Measured	10	1	0
Field Measurements	Temperature	Water	YSI-Field Measured	10	1	0
	Turbidity	Turbidity Water Nephalometer Field Measure		10	1	0
	Nitrate	Water Hach Kit-Field Measured		10	1	0
	Sulfate	Water	Hach Kit-Field Measured	10	1	0
	Ferrous Iron	Water	Hach Kit-Field Measured	10	1	0
Field	Alkalinity	Water	Water Hach Kit-Field Measured		1	0
Measurements (cont.)	Dissolved Manganese	Water	Water Hach Kit-Field Measured		1	0
	Methane	Water	RSK 175	10	3	0 ²
	BTEX	Water	EPA method 8260	10	1	1
Laboratory	GRO	Water	AK101	10	1	1
Analyses	DRO/RRO	Water	AK102/AK103	10	1	1
	PAHs	Water	8270C-SIM	10	1	1
	PCBs	Water	EPA 8082	10	1	1
	Metals-RCRA 8	Water	EPA 6010/6020	10	1	1

Table 3-7 MOC Groundwater Monitoring Samples

Notes:

¹MS/MSDs will be collected for every batch (less than or equal to 20 samples) submitted to the laboratory.

²The laboratory has not approved method for spiking ethane samples in water.

AK	=	Alaska Method	MS	=	matrix spike
BTEX	=	benzene, toluene, ethylbenzene, and xylenes	MSD	=	matrix spike duplicate
DRO	=	diesel range organics	PCBs	=	polychlorinated biphenyls
EPA	=	U.S. Environmental Protection Agency	QC	=	quality control
GRO	=	gasoline range organics	RCRA	=	Resource Conservation and Recovery Act
MOC	=	Main Operations Complex	RRO	=	residual range organics

3.6 DRO DELINEATION AND CHARACTERIZATION AT THE MOC

An Ultraviolet Optical Screening Tool (UVOSTTM) with laser-induced fluorescence (LIF) will be used for determining the nature and extent of contaminants at the MOC (Sites 10, 11, 13, 15, 17, 19, 20, and 27). Soil samples will be collected to correlate the UVOST results. The estimated number of UVOST screening locations is 200 from various areas in the MOC shown on Figure 16. Sixteen DRO correlation soil samples will be collected from the UVOST area. Four DRO soil samples will be taken from high LIF areas, 4 samples from moderate LIF areas, 4 samples from low LIF areas, and from 4 non-detect sample locations. The soil correlation samples will be collected over the entire UVOST investigative area. The UVOST screening and sample quantities may be altered in the field with consultation with the QAR to meet the objectives as described above. All holes created during this investigation will be immediately sealed with dry #8 volclay/bentonite and temporarily labeled with pin flags.

3.6.1 UVOST Operation

The UVOST investigation will be performed using LIF technology employed by the UVOST system, which provides continuous, real-time data of potential petroleum-contaminated soil. A Geoprobe[®] 6610DT direct-push, track-mounted probe will be used to advance the probes that contain the UVOST system. A xenon helium hydrogen chloride excimer laser (308-nanometer wavelength) is used as the energy source. Ultraviolet light from the laser is transmitted through a silicon-clad optical fiber that terminates at a sapphire window. The sapphire window is in contact with the subsurface soil. The UVOST system can detect non-chlorinated, multi-ring, polynuclear aromatic hydrocarbons (PAHs) (fuel) in saturated and unsaturated soils. If petroleum hydrocarbons are present, the laser light excites the PAH fraction into releasing energy as fluorescence. The intensity of the fluorescence is used as an indicator of the relative contaminant concentration. The LIF results are acquired and displayed in real time with depth in a fluorescence vs. depth (FVD) log.

The LIF signal response is optimized using a proprietary reference solution, M1, from Dakota Technologies, Inc., before each UVOST push. The UVOST software package allows for analysis of the amount of fluorescence at each of the four different response wavelengths that

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make up the LIF reading. The amount of fluorescence at each of the four wavelengths is called the "fuel signature." The LIF data is reviewed by the field team to identify the concentration and fuel type (gasoline range organics [GRO], DRO, and RRO) of the contaminant. This information will be used to determine optimal locations for further investigation points.

Naturally occurring fluorescent minerals, such as carbonates, can yield false positives. Data from sampling and laboratory analysis of soil samples will assist in determining if false positives have occurred. False negatives may occur where the DRO/GRO concentrations are near the limit of detection for the LIF probe, which is approximately 100 mg/kg.

The following is a list of operations performed to ensure data quality:

- Monitor the wave pattern on the oscilloscope.
- Verify that the M1 signal level and the time delay are in the proper position and within limits.
- Calibrate the UVOST with M1 prior to every push.
- Monitor the graphic output on the FVD log.
- After every push, place M1 on the probe window to visually verify that the signals are within tolerance.

Exaggerated background readings or loss of signal from the probe are the usual indications that a problem has occurred. Typical problems include:

- Cracked window,
- Dust on the mirror,
- Software errors, and
- Electronic malfunctions.

When system errors occur while pushing the probe, that location is probed again until a useable dataset is acquired.

3.6.2 Correlation Sampling

The objectives of the soil sampling and field-screening analysis associated with the UVOST investigation is for correlation. The correlation will assess the potential for false positive or false negative results produced by the UVOST investigation, and establish a qualitative correlation between LIF results and the chemical parameters. The UVOST correlation samples are collected from soil intervals showing highly contaminated, moderately contaminated, slightly contaminated, and uncontaminated readings. The soil samples will be visually classified to identify soil type and observations about petroleum contamination, such as oil and staining, and will be documented on field sheets.

Correlation samples will be collected from discrete depth intervals and analyzed for DRO by AK102. A minimum of 16 samples shall be identified over a range of contaminant concentrations within the UVOST study area. Sample locations will be decided in the field. Four samples will be collected from areas with high LIF responses; 4 samples from areas with moderate LIF responses; 4 samples from areas with low LIF responses, and 4 samples from areas with non-detect LIF responses.

Sampling and Analysis Plan Contract No. W911KB-10-C-0002

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Sampling and Analysis Plan Contract No. W911KB-10-C-0002 NE Cape HTRW Remedial Actions Bristol Project No. 410026

4.0 FIELD SAMPLING PLAN

The following sections detail the sampling procedures for the project. Areas will be sampled for PCB-, POL-, and arsenic-contaminated soil. Field-screening and confirmation samples will be collected from sites following excavation to ensure that no contamination remains above site cleanup levels. Bulk bags used to containerize the contaminated soil will be sampled for waste characterization. Waste characterization is required for transport and disposal at the disposal facilities.

Table 4-1 summarizes the analytical methods, containers, and holding times for samples collected at NE Cape in 2010.

Matr	ix	Parameter Analytes	Analytical Method	Container Description (Minimum)	Preservation/Holding Time
Soil DRO/RRO		AK 102/103	8-oz wide-mouth, clear glass jar, TLC	14 days to extraction/ 40 days to analysis	
Soi	I	RCRA Metals (Arsenic)	EPA 6020	8-oz wide-mouth, clear glass jar, TLC	180 days
Soi	I	PCBs	EPA 8082	8-oz wide-mouth, clear glass jar, TLC	14 days to extraction/ 40 days to analysis
Soi	I	Total Organic Carbon	EPA 9060	4-oz wide-mouth, clear glass jar, TLC	Unpreserved, Cool 4° ± 2°C / 28 days to analysis
Wate	er	Methane	RSK 175	3 40-mL amber VOA vials	14 days to extraction and analysis
Wate	er	GRO/BTEX	AK 101/ EPA 8260B	6, 40-ml VOA vials	HCl, Cool 4° ± 2°C / 14 days to analysis
Wate	er	PCBs	EPA 8082	1-Liter amber glass	Cool 4° ± 2°C / 14 days to extraction/40 days to analysis
Wate	er	RCRA 8 Metals	EPA 6010/6020	Plastic, 250 mL	nitric acid, Cool 4° ± 2°C 6 months to extraction and analysis
Notes: °C	=	degrees Celsius		mL = milliliters	
AK	=	Alaska Method		PCBs = polychlorina	ated biphenyls
BTEX	=	benzene, toluene, ethyl	benzene, and xylenes		Conservation and Recovery Act
DRO				nge organics	
EPA	=	U.S. Environmental Pro			ed screw cap
GRO	=	gasoline-range organics	3	VOA = volatile orga	anic analysis
HCI	=	hydrochloric acid			

Table 4-1 Sample Collection, Preservatives, and Holding Times

4.1 SOIL SAMPLING PROCEDURES

The procedures outlined below will be used for the collection of field screening and confirmation soil samples. Field-screening samples will be collected in resealable plastic bags (Ziplocs[®]) that will be analyzed by an on-site, field-screening laboratory. Confirmation samples will be collected in clean glass containers provided by TestAmerica and shipped to the TestAmerica laboratory in Tacoma, Washington.

The POL excavations will be sampled according to the Draft DEC Field Sampling Guidance (ADEC, 2010), which directs the collection of two primary grab samples for the first 250 square feet of floor or sidewall, and one sample for each additional 250 square feet. PCB excavations will be sampled according to TSCA regulation 40 CFR Part 761, which directs the collection of one primary grab sample for every 25 square feet of floor or sidewall. Regardless of size of excavation a minimum of 3 samples from each excavation, including sidewalls, will be collected and analyzed by the field screening and confirmation laboratories. Soil samples for field screening and confirmation samples will be collected from the excavated areas using sample grids. Soil for field-screening and laboratory confirmation samples will be collected at each site is dependent on the size of the excavation and the contaminant of concern. The field sampling crew will establish 25-foot by 25-foot sampling grids in the DRO/RRO and arsenic work areas and 5-foot by 5-foot grids in the PCB work areas.

Discrete soil samples will be collected using a clean, stainless-steel spoon, trowel, or a clean, nitrile-gloved hand. The following sampling procedures will be used for discrete soil sample collection:

- 1. Determine the location and number of samples required, as presented in Section 3.0. The sample locations will be from areas of highest contamination, based upon historical results, field screening, and observations.
- 2. Soil field-screening and confirmation samples will be comprised of discrete grab samples.
- 3. For PCB samples a maximum of nine discrete samples will be composited. Composited samples will be from contiguous sample points from adjacent grids from

the same level (depth below ground surface). Sidewall samples may comprise one composite sample, but floor and sidewall samples may not be composited in the same sample. Primary confirmation samples will be composited by the laboratory and individual samples selected for each composite will be clearly identified on the chain-of-custody (CoC) form.

- a. If a PCB composite sample made up of nine discrete samples contains PCBs above 0.11 mg/kg, the results suggest that a single discrete sample is above the 1.0 mg/kg cleanup, and, therefore, all discrete samples for this PCB composite sample will be analyzed by either the field-screening laboratory or the fixed-base laboratory.
- b. At times, the field samplers may collect a PCB composite sample made up of less than nine discrete samples to keep the samples contiguous and at the same level. If a PCB composite sample is made up of five discrete samples and the analytical result is above 0.2 mg/kg PCB, all five discrete samples making up this PCB composite sample will be analyzed.
- 4. Label appropriate glassware or bulk bag in accordance with Section 4.5.
- 5. If the excavation depth is less than 4 feet and the sidewalls are deemed stable, discrete samples will be collected directly from native soils exposed on the floor and sidewalls of the excavation. Samples collected for DRO/RRO analyses must come from freshly exposed surface locations or from subsurface locations if the area has been exposed for more than 6 hours. Samples collected for PCB or arsenic analyses may be collected from the excavations or surface with no time limit due to the lack of volatility of PCBs and arsenic.
- 6. If the excavation depth is greater than 4 feet or the sidewalls are deemed unstable, the excavator bucket will be used to collect native soils from the desired locations as described in Section 4.1.1. Care will be taken to minimize the amount of soil taken from the floor or sidewall of the excavation when sampling.
- 7. Place soils directly into sampling containers with a disposable sample spoon or gloved hand.
- 8. Record the sampling grid, sample depth and location, date, time, and analysis to be conducted in the field logbook.
- 9. Secure container lids and place samples in an iced cooler.
- 10. Record sampling information, including date, time, analysis to be conducted, and sampling location on the CoC form.
- 11. Decontaminate sampling equipment in accordance with Section 4.7, and change sampling gloves between each sampling locations.

4.1.1 Sampling from an Excavator Bucket

If any excavation depth is greater than 4 feet or the sidewalls are deemed unstable, the excavator bucket will be used to collect native soils from desired locations for either field

screening or confirmation samples. The procedures for collecting a sample from the bucket

are the following:

- 1. If excavator buckets are not cleaned between sample locations at a work area, collect samples from material in the center of the bucket.
- 2. Prior to collecting samples from soil in the excavator bucket, dress the surface with a stainless steel shovel, spatula, knife, or spoon to remove at least six inches of surface layer of soil, which may have been smeared across the trench wall as the bucket passed.
- 3. Be aware of "slough" material in the bucket that is not representative of the specified sample depth.
- 4. Collect the sample using a stainless steel spoon or gloved hand. Exclude any large rocks or foreign material.
- 5. Record the sample ID, location (LocID), date, time, and requested analyses for the sample in a field logbook.

4.1.2 Sampling Soil from Bulk Bags

A sub-sample will be collected from each DOT-approved bulk bag and will be composited with as many as seven other grab samples to make one composite sample. The sub-sample will consist of soil collected from each end of the container (two total per bulk bag). Approximately 8 ounces of soil will be collected from the two locations using a clean stainless steel trowel, and then placed into a stainless steel bowl for compositing. The samples will be collected from a depth of approximately one foot below the top soil layer in the bag. Once the soil is thoroughly mixed, the sample containers will be filled and labeled. The sample number and its associated containers will be immediately logged in the field book.

Bulk PCB and POL waste sample composites will be submitted to the field-screening laboratory for waste characterization. The disposal facility will accept field-screening results for disposal purposes.

4.1.3 Soil Sampling from a Macro-Core[®] Sampling System

The samples collected during the UVOST investigation will be collected using a Geoprobe 6610DT direct-push, track-mounted probe system with a Macro-Core sampler. Soil samples will be collected at depths determined by the UVOST screening results for correlation

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sampling. The Macro-Core will be driven to the depth determined for the desired LIF response. The Macro-Core sampler is 4-feet in length and 1.5 inches in diameter. The drillers will line up the midsection of the core to the desired depth of the sample. The core will be split open, and samples retrieved at the midsection of the core

The soil will be collected for DRO analysis from the split plastic sleeve using a clean stainless steel spoon, disposable sample spoon, or by hand wearing a fresh pair of nitrile gloves for each sample. Prior to soil sample collection for DRO analysis, the LIF response of the sample will be measured ex-situ. Once the LIF response has been measured, the soil will be placed in a laboratory-provided container (not packed), then placed in a chilled sample cooler. For duplicate samples, the soil will be placed in a clean stainless steel mixing bowl and homogenized. After which, one container will be filled for the duplicate sample and another container filled for the primary sample.

4.2 FIELD SCREENING

4.2.1 PID Field Screening

A PID may be used to identify POL-contaminated soil in the different work areas and determine field-screening and confirmation sample locations. Field-screening and confirmation samples will be obtained from locations where in-situ PID readings and observations indicate the most heavily contaminated or from the center of the sampling grid.

In-situ PID screening may occur at the surface or within an excavation, and it is a fairly rapid procedure where the screener uses a rock hammer to create an indent in the excavation floor or wall and then immediately places the PID tip in the indent to obtain a reading. In-situ screening is best suited to confirm presence or absence of POL and also to locate hot spots in a relatively small area.

4.2.2 On-Site Field Screening Laboratory

An on-site field-screening laboratory will utilize gas chromatographs to provide results for DRO/RRO analyzed by Method AK 102/103 and for PCBs analyzed by EPA Method 8082. The field-screening laboratory will not be certified. The SOPs for the field screening laboratory are in Appendix C.

Results from the on-site field screening laboratory will be used to direct excavations, UVOST correlation soil samples, and characterize waste, but will not be used to confirm that cleanup goals have been achieved at the sites.

4.3 WATER SAMPLING PROCEDURES

The procedures outlined below will be used for the collection of water samples. Surface water samples will be collected at Site 8, Site 9, and possibly at Site 6 near MW 6-1. Groundwater samples from 10 monitoring wells will be collected at the MOC if the wells have not been compromised by frost-jacking. Water samples that are not field analyzed will be shipped to TestAmerica in Tacoma, Washington.

4.3.1 Surface Water Sampling

The in-situ surface water field parameter data, including pH, DO, conductivity, temperature, and ORP will be collected using a YSI water quality meter with a flow through cell. Turbidity will be measured using a Hach 2100P turbidimeter.

In order to avoid adding turbidity to the surface water samples at Site 8, the most downgradient Decision Unit sample points will be sampled first, followed by the middle Decision Unit sample points, and finally, the uppermost Decision Unit sample points. The eight randomly selected points from each Decision Unit will be individually measured for turbidity, pH, DO, conductivity, temperature, and ORP. All measurements and locations will be recorded in the field notebook. Water samples will be collected from each of the eight sample points in each Decision Unit for methane, nitrate, sulfate, ferrous iron, alkalinity, and dissolved manganese. The surface water samples will be collected by dipping a clean unpreserved 1.0-liter sample bottle in an undisturbed water location, then gently filling the containers for methane analysis by the off- site laboratory. The clean unpreserved sample bottle will then be filled for use in measuring nitrate, sulfate, ferrous iron, alkalinity, turbidity, and dissolved manganese in the field laboratory using a Hach DR890 spectrometer and the appropriate reagents for each test.

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4.3.2 Water Level Measurement

Prior to groundwater sample collection, the monitoring wells selected by the USACE at the MOC site will be opened and a water level measurement collected. The following procedures will be followed for measuring the water levels:

- Monitoring wells will be assessed for condition and frost-jacking. Wells that are in a condition to be sampled will be opened and the cap will be removed from the well. In order to allow time for the well to equalize, the well will not be measured for at least one hour. To accomplish this efficiently, all monitoring wells will be opened and well caps removed, after which the water level in each well will be measured in the order that the wells were opened.
- If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing) one will be made. The location of the reference mark will be described in the field notebook.
- The water level in each monitoring well will be measured from the top of the casing to the water level, to the nearest 0.01 foot. The measurement will be recorded in a field notebook;
- The total depth of the well will be measured and recorded in the field notebook;
- The water level tape will be cleaned with Alconox[®] soap and tap water, and rinsed with deionized water between each use;
- The cap will be placed back on the well after the water level is measured and recorded.

4.3.3 Groundwater Sampling

Groundwater samples at the MOC will be collected from up to 10 monitoring wells and analyzed for BTEX, PCBs, GRO, DRO, metals, PAHs, and MNA parameters. The objective of the groundwater sampling is to establish a comparison baseline outside the anticipated future excavation areas at the MOC.

The groundwater sampling procedure involves using a peristaltic pump with ¹/₄-inch or 3/₈inch inside diameter polyethylene tubing to purge the well. Although the well recharge rates are not available, the peristaltic pump should be able to maintain a very low pumping rate. The methodology for groundwater purging and sampling is summarized below:

1. After the water level and total depth have been measured, lower the tubing slowly (to minimize disturbance) into the well to the bottom of the well and then lift it about a foot up from the bottom.

- 2. Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs.
- 3. During well purging, monitor indicator field parameters every three to five minutes. The following field parameters will be monitored: turbidity, temperature, specific conductance, pH, ORP, and DO. All measurements, except turbidity, will be obtained using a YSI meter attached to a flow-through cell. A transparent flow-through cell will be used. This allows the field personnel to watch particulate buildup within the cell. If the cell needs to be cleaned during purging, continue pumping and disconnect the cell for cleaning, then reconnect and continue monitoring.
- 4. Groundwater samples can be collected after three casing volumes have been removed and the field parameters stabilize within the following limits:
 - Turbidity (10% for values greater than 1 nephelometric turbidity units [NTU]),
 - DO (10 %),
 - Specific conductance (3%),
 - Temperature (3%),
 - pH (0.1 units),
 - ORP (10 millivolts).
- 5. Do not increase the pump rate while sampling. Groundwater samples will be collected from water before it has passed through the flow-through cell (use a by-pass assembly or disconnect the cell). BTEX and GRO samples will be collected first by pumping directly into pre-preserved sample containers. The sample containers will be filled by discharging the flow gently down the inside of the container with minimal turbulence. After the BTEX/GRO sample containers have been filled, fill all other sample containers without shutting off the pump.
- 6. Groundwater samples for MNA parameters will be analyzed in the field laboratory using a Hach field kit for nitrate, sulfate, ferric iron, dissolved manganese, and nitrate. A clean1-liter unpreserved sample container will be used to collect water for field analysis using the Hach field kits.

In the event that after following the procedures outlined above, the well dewaters during purging, the groundwater samples will be collected as soon as the water level has recovered sufficiently to collect the appropriate volume needed.

The pump tubing will be replaced between each well. All water generated during purging will be treated through granular activated carbon filtration.

4.4 SEDIMENT SAMPLING PROCEDURES

Discrete sediment samples will be collected using a clean, stainless-steel spoon, trowel, or a clean, nitrile-gloved hand. Sediment samples will be collected after all surface water samples have been collected. The following sampling procedures will be used for discrete soil sample collection:

- 1. The eight sediment sample locations within each of the three DUs will be located as described in Section 3.4.
- 2. One clean, resealable plastic bag will be half-filled from sediment at each of the eight locations. After the container is filled with sediment, water, if present, will be poured out of the bag. Once the eight bags of sediment have been collected, they will be combined in a large stainless steel mixing bowl.
- 3. The sediment will be composited by mixing, using a clean stainless steel spoon. Once the sediment is thoroughly mixed, a clean 8-ounce sample container will be filled.
- 4. A duplicate sample will be collected by dividing the original homogenized sediment (from the eight sample points) into two sections. The duplicate will be collected from one of the sections, and the primary sample from the other section.
- 5. Record the sampling grid, sample depth and location, date, time, and analysis to be conducted in the field logbook.
- 6. Secure container lids and place samples in an iced cooler.
- 7. Decontaminate sampling equipment in accordance with Section 4.7, and change sampling gloves between each sampling location.

4.5 SAMPLE DESIGNATIONS

The samples will be numbered as follows: ##NCXXMMZZ, where ## is the year, NC indicates NE Cape, XX is the site identifier, MM is the sample type, and ZZ is the sample number. See Tables 4.2 and 4.3 for site identifier and sample type information. Field QC samples will be labeled and numbered in the same manner to prevent the laboratory from distinguishing them from other site samples.

Trip blanks will be included in sample shipments (SDGs) that contain GRO or VOC samples. Trip blanks will be identified with the date precluding the designation "trip blank". The first shipment of the day will have a -1 after trip blank, -2 for the second shipment etc. An example of a trip blank ID is 0701trip blank-1. This indicates the samples were shipped on July 1st in the first SDG prepared for shipment.

Site Identifier	Site Name
01	Site 1-Airstrip
03	Site 3-Cargo Beach Pumphouse
06	Site 6 Cargo Beach Road Drum Field
08	Site 8-POL Pipeline Break
13	Site 13-Power Plant
16	Site 16-Paint Dope Storage Building
21	Site 21-Wastewater Treatment Plant
28	Site 28-MOC Drainage Ditch
31	Site 31-White Alice Communications Station
32	Site 32-Lower Tram

Table 4-2 Sample Site Identifier (XX in the sample number)

 Table 4-3
 Sample Types (MM in the sample number)

Sample Type	Sample Designation
SB	Soil or Sediment
BW	Bulk Waste (Containerized Soil Characterization)
WA	Water

4.6 QUALITY CONTROL SAMPLES

Quality control samples will be collected as duplicate samples for field duplicates and MS/MSDs. A discussion of each QC type is provided below and detailed in the QAPP. Matrix spike/matrix spike duplicate samples are not required for waste characterization samples. Field duplicates are required on waste characterization samples at a 10 percent frequency.

4.6.1 Field Duplicates

Field duplicate/split samples will be collected as indicated in Section 3.0. If additional sampling is required, the QAR will be notified for approval. Quality control samples will be collected at a rate of 10 percent. The duplicate sample will be collected at the same location as the environmental sample, at the same time the environmental sample is collected.

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4.6.2 Matrix Spike/Matrix Spike Duplicates

A minimum of one MS and one MSD sample shall be analyzed for every sample batch in soil. No MS/MSD samples will be collected from waste characterization samples. Matrix spike/matrix spike duplicate quantities will be collected as indicated in Section 3.0 and Table 3-3.

4.7 DECONTAMINATION PROCEDURES

Disposable sampling equipment will be used when possible. Pre-cleaned sample containers will be provided by the analytical laboratory. Non-disposable field sampling equipment and heavy excavation equipment will be decontaminated as follows:

- Trowels or spoons used for soil sampling will be scraped clean of gross contamination and washed in an Alconox solution, followed by potable and deionized water rinses.
 - If temperatures are near or below 32 degrees Fahrenheit, all equipment will be drybrushed to remove contamination.
- Sampling equipment will be allowed to air dry before reuse.
- Plastic sheeting will be placed beneath the bucket of any heavy piece of equipment prior to being swept clean with brushes and brooms to remove any contaminated soil from previous locations.
- Soil, plastic, and fluids generated during sampling equipment decontamination activities will be added to contaminated soil in the bulk bags for disposal.

4.8 **DOCUMENTATION**

Field personnel will document administrative and technical activities. Examples of the forms discussed in this section are presented in Appendix D. The following types of documentation will be required:

- Field logbooks,
- Sample record log,
- Photograph logs,
- Sample labels,
- CoC records;
- Custody seals,

- Receipt of Sample Forms, and
- Sample Summary Forms.

4.8.1 Field Logbooks

Field logbooks will be used to document field and sampling activities. Each logbook will include the following information for each sample:

- Date and time,
- Site/sampling location,
- Sample identification number,
- Analytical methods, jar types, and preservatives used,
- Drawing of the site and sampling location, and
- Comments.

Each page of the field logbook will be pre-numbered, dated, and signed by the author. The field logbook will be sturdy, weatherproof, and bound. All writing will be done in waterproof, black, permanent ink. No pages will be removed. Blank pages will be marked "page intentionally left blank." Mistakes will be crossed out with a single line, initialed, and dated.

4.8.2 Sample Record Log

The Environmental Samplers will maintain a sample record log containing the following:

- Sample number,
- Sampling location [Location ID (LOCID)],
- Date and time of sample collection,
- Sample matrix,
- Sample depth (if appropriate),
- Sampler name,
- PID Field screening result,
- Analyses requested and preservative used (where applicable),
- Type of sample (project or duplicate),
- MS/MSD information,

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- CoC number,
- Sample shipping date
- · Date analytical results were received, and
- Comments.

4.8.3 Equipment Calibration Logs

Field screening instruments (water quality meter, PID, and gas chromatographs) will be calibrated each day before use and recalibrated in the event of extreme climate changes. Daily calibration checks are acceptable if the calibration is within the manufacturers stated limits or within method specific calibration criteria for gas chromatographs. All calibration information will be included in the field notes or instrument logbook. For each piece of fieldscreening equipment, the following documentation will be made in the field notebook:

- Instrument,
- Date and time,
- Calibration results, and
- Signature of person conducting the calibration.

4.8.4 Photograph Logs

Photographs will be taken in the field to document sampling locations and conditions. Each photograph log will contain the following:

- Date and time,
- Location,
- Photograph description,
- View of direction, and
- Photographer/comments.

4.8.5 Sample Labels

Labels are required for analytical samples. Sample IDs will follow the procedures described in Section 4.6. Site- and time-dependent information will be added to the labels using indelible ink. The labels will be protected from water and solvents with clear label protection tape and will contain the following:

- Project name,
- Date and time of collection,
- Sample number,
- Analysis to be performed,
- Preservative (if applicable), and
- The sampler's name.

4.8.6 Chain-of-Custody

The CoC forms will document the sample collection and analysis required for each sample. The history of each sample and its handling will be documented from collection through transference to the analytical laboratory. Internal laboratory records will document custody of the sample from the time it is received through its final disposition. The CoC form will be filled out after the samples have been collected and will contain the following information:

- Project Name;
- Quote number (TestAmerica Quote Number 58003534);
- North Pacific Division Laboratory (NPDL) Number 09-034;
- Location identification (LOC ID) = "001-01, 001-02, etc." (samples from different locations will not be mixed on CoCs), (Waste characterization Samples from similar sources may be mixed, but not composited together.);
- Names of samplers;
- Samplers' signatures;
- Sample identification;
- Date and time sample was collected;
- Number of containers per sample;
- Sample matrix;
- Preservative (if applicable); and
- Analysis requested.

4.8.7 Custody Seals

Custody seals will be signed and dated by the personnel preparing the seals. Two seals will be attached to the lid and body of each cooler. The seals will be affixed to the cooler, so that the containers cannot be opened without breaking the seals.

4.8.8 Receipt of Sample Forms

Upon receipt of analytical samples, the laboratory will complete and transmit to the field site a Receipt of Sample Form, which will include the sample numbers received, the requested analyses, and any corrective actions deemed necessary. This form will allow field personnel to ensure that all required analyses are conducted on each sample and to document any sample shipping problems. A copy of each Receipt of Sample Form will be kept as a part of the project record.

4.9 SAMPLE PACKAGING AND SHIPPING

All analytical samples will be shipped in accordance with International Air Transport Association 2.7, Dangerous Goods in Excepted Quantities, by charter aircraft from NE Cape to Nome, Alaska, and then transported via Alaska Airlines Goldstreak package service, to TestAmerica-Tacoma. Analytical samples will be packaged in the following manner:

- 1. Place each sample in a plastic Ziploc bag and seal.
- 2. Place frozen ice packs on the bottom of the cooler.
- 3. Wrap individual samples in bubble wrap and place in the cooler, or place individual samples into the cooler and fill empty spaces with newspaper.
- 4. Cover headspace inside the cooler with frozen ice packs.
- 5. Place the CoC form inside a sealed Ziploc bag and tape it to the inside cover of the cooler.
- 6. Notify the laboratory of approximately when and how many samples will arrive. Upon receipt at TestAmerica, a copy of the CoC form and cooler receipt form will be e-mailed to cooler.receipt@usace.army.mil within 24 hours of sample receipt.

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5.0 QUALITY ASSURANCE PROJECT PLAN

This QAPP will be used for the HTRW removal activities at NE Cape. Changes to this QAPP will follow Bristol corporate policy and procedures, which require that if significant changes are required, they be included in a revision to this document. The USACE Contracting Officer, or designee, and the ADEC will be provided with the opportunity to comment on and approve revisions.

This QAPP is based generally on requirements and guidance provided by the following:

- Engineering Requirement (ER) 1110-1-263, Engineering and Design Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities (USACE, 1998a);
- EM 200-1-2, Technical Project Planning (TPP) Process (USACE, 1998b);
- EM 200-1-3, Engineering and Design Requirements for the Preparation of Sampling and Analysis Plans, (USACE, 2001);
- EM 200-1-6, Environmental Quality Chemical Quality Assurance for Hazardous, Toxic, and Radioactive Waste (HTRW) Projects (USACE, 1997);
- U.S. Environmental Protection Agency (EPA)/QA/R-5, EPA Requirements for Quality Assurance Project Plans (EPA, 2001);
- EPA/QA/G-9, Guidance for Data Quality Assessments/Practical Methods for Data Analysis (EPA, 2000); and
- Quality Systems Manual, version 4.1 (DoD, April, 2009).

The structure of this QAPP follows basic USACE guidance for preparation of QAPPs, which is adapted from guidance in EPA Requirements for Quality Assurance Project Plans (EPA, 2001). A glossary of terms is presented as Appendix E.

5.1 **DEFINITIONS AND OBJECTIVES**

The QAPP is defined as the total integrated program for ensuring reliability of monitoring and measuring data. The QC is defined as the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measuring process.

The objectives of the QA program are to ensure that 1) the procedures used will not detract from the quality of the results, and 2) all activities, findings, and results follow the terms and conditions of this QAPP, and are documented.

5.2 DATA QUALITY OBJECTIVES

5.2.1 Field-Screening Data

The DQOs for field screening shall be to obtain relatively rapid, reasonably accurate field data sufficient to 1) support field decisions, 2) segregate and dispose of wastes based on their waste category, and 3) to determine if potential RCRA, TSCA, or DOT wastes are present. Field-screening data will be used to support the removal actions.

Field measurement QA objectives will be addressed as follows:

- All field instruments will be calibrated in accordance with the manufacturer's instructions and/or the associated implemented SOPs to address accuracy;
- Precision will be addressed by taking replicate measurements and comparing these measurements to manufacturer's specifications for the individual instrument or analytical method;
- Representativeness will be based on professional judgment and examination of the matrix from which the sample was measured and/or collected;
- The completeness goal is 90 percent, based on the proposed number of measurements compared to the number of completed measurements; and
- Samples will be considered comparable if the instrument is functioning within the procedure specifications and if calibrations are made within the recommended frequency, as specified by the manufacturer or the implemented SOP.

5.2.2 Laboratory Analytical Data

Data from laboratory analysis of site samples will be used to confirm that all soil in place meets site cleanup goals. All the fixed laboratory chemical data generated under this contract shall comply with:

- The most recent version of the DoD Quality Systems Manual for Environmental Laboratories (DoD, 2009);
- Alaska Department of Environmental Conservation, 18 AAC 75 Oil and Other Hazardous Substances Pollution Control (revised as of October 9, 2008); and
- Any applicable federal, state, or local requirements.

The QA objectives for laboratory analytical data are defined below.

5.2.2.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Precision is expressed in terms of relative percent difference (RPD). The RPD is calculated as follows:

$$RPD = (S - D) \times 100$$
[(S+D)/2]
Where: S = initial sample result
D = duplicate sample result

The laboratory objective for precision is to equal or exceed the precision demonstrated for similar samples and to ensure that the RPD falls within the established control limits for the methods. In general, the LCS/LCSD, the MS (initial spiked sample result) and MSD (duplicate spiked sample result), and blind duplicates will be used to determine the precision. The precision objectives for LCS/LCSD and MS/MSD samples are presented in Table 5-1. The LCS/LCSD results will be the primary method of measuring batch precision. The MS/MSD and blind duplicates will be used to measure field precision and support of batch precision. When sample results are greater than 4 times to concentration of the matrix spike, accuracy and precision will be evaluated and reported in the data verification summary, however, sample results will not be flagged due to out-of-control recoveries or precision. Similarly, field duplicates with reported concentrations less than the reporting limit will be evaluated and reported in the data verification summary, however, sample results will not be flagged due to RPD values exceeding the project control limits due to the lesser degree of accuracy below the reporting limit. The field duplicate precision acceptance criteria will follow the ADEC recommended limits of 30% RPD for water samples and 50% RPD for soils and waste.

Analyte	LOQ (µg/kg)	Criteria (µg/kg)	Surr. %R	LCS/LCSD Control Limits	LCS/LCSD RPD Limit	MS/MSD Control Limits	MS/MSD RPD Limit
AK 102/AK 103							
Diesel range Organics	20,000	9,200,000		75-125	20	50-150	20
Residual range Organics	50,000	11,000,000	_	60-120	20	60-120	21
Surrogates							
o-Terphenyl			50-150	60-120			
n-Triacontane-D62			50-150	60-120			
RCRA Metals - 6010B/6020			代本品的				
Arsenic	3,000	3,900		80-120	35	75-125	35
Barium	500	1,100,000		80-120	35	75-125	35
Cadmium	500	5,000		80-120	35	75-125	35
Chromium	1,300	25,000		80-120	35	75-125	35
Lead	1,500	400,000		80-120	35	75-125	35
Silver	1,000	11,200		80-120	35	75-125	35
EPA 8082							
Aroclor [®] 1016	10	1,000		40-140	20	40-140	20
Aroclor1221	10	1,000					
Aroclor 1232	10	1,000					
Aroclor 1242	10	1,000					
Aroclor 1248	10	1,000					

Table 5-1 LOQs and QC Acceptance Criteria for Soil

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Table 5-1	LOQs and QC Acceptance Criteria for Soil (continued)

Analyte	LOQ (µg/kg)	Criteria (µg/kg)	Surr. %R	LCS/LCSD Control Limits	LCS/LCSD RPD Limit	MS/MSD Control Limits	MS/MSD RPD Limit
EPA 8082						The second second	
Aroclor 1254	10	1,000					
Aroclor 1260	10	1,000		60-130	20	60-130	20
Surrogates							
Tetrachloro-m-xylene			45-155				
Decachlorobiphenyl			60-125				

Notes:

Acceptance criteria from TestAmerica, Tacoma (also provided in Appendix F)

-		=	not applicable	MS	=	matrix spike
%	R	=	percent recovery	MSD	=	matrix spike duplicate
%	RPD	=	relative percent difference	LOQ	=	Limit of Quantitation
μg	/kg	=	micrograms per kilogram	QC	=	quality control
Ak	<	=	Alaska Method	RCRA	=	Resource Conservation and Recovery Act
EF	PA	=	U.S. Environmental Protection Agency	RPD	=	relative percent difference
LC	s	=	laboratory control sample	surr.	=	surrogate

LCSD = laboratory control sample duplicate

5.2.2.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. For volatile organic compounds, PCBs, and semi-volatile organic compounds, surrogate compound recoveries are used to assess accuracy and method performance for each sample analyzed. Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The LCS and LCSD results are the primary tool for measuring accuracy. The formula for calculation or accuracy is included in Table 5-2 as percent recovery (% R) from laboratory pure water and sample matrices, and is as follows:

	%	R =	(<u>SSR - SR</u>) x 100
			SA
Where:	SSR	=	spike sample result
	SR	=	sample (unspiked) result
	SA	=	spike added

The accuracy objectives for surrogate recoveries, and laboratory control samples are presented in Table 5-4.

5.2.2.3 <u>Representativeness</u>

Representativeness is the degree to which the sample data accurately and precisely represent an environmental condition. Representativeness will be satisfied by ensuring that sampling locations are selected properly (i.e., represent the range of environmental conditions observed) and an adequate number of samples are collected, as presented in Section 4.0. Field replicates will be used to assess representativeness.

5.2.2.4 Completeness

Completeness is the percent of measurements that are judged to be valid. The completeness of the data means that all the required samples have been collected and requisite analyses performed to generate an adequate database to successfully complete the remedial design

studies. The completeness goal shall be 90 percent for the analytical procedures, as described in Section 5.6. Completeness will be determined by comparing the number of analyses attempted against the number of subsequent data points judged to be usable for the designated purpose(s).

5.2.2.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another. The FSP (Section 4.0) specifies the sampling method to be employed, the CoC methods to be used for the transfer of the samples to the analytical laboratories, and the analytical techniques to be implemented at the laboratories.

Statistic	Symbol	Formula	Definition	Uses
Mean	x	$\frac{\begin{pmatrix} n \\ \Sigma & x_{i} \\ i=1 \end{pmatrix}}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left(\frac{\Sigma(x_1-\overline{x})^2}{(n-1)}\right)^{\frac{1}{2}}$	Measure of relative scatter of the data	Used to calculate variation of measurements
Relative Standard Deviation	RSD	(S/X) x 100	Relative standard deviation adjusts for magnitude of observations	Used to assess precision for replicated results
Percent Difference	%D	$\frac{x_1 - x_2}{x_1}$ x 100	Measure of the difference of two observations	Used to assess precision
Relative Percent Difference	%RPD	$\left(\frac{(X_1 - X_2)}{(X_1 + X_2)/2}\right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess total and analytical precision of duplicate measurements
Percent Recovery	%R	$\left(\frac{X_{meas}}{X_{true}}\right)$ x 100	Recovery of spiked compound in pure matrix	Used to assess accuracy
Percent Recovery	%R	value of spikedvalue of unspiked samplevalue of samplesampleValue of added spike 100x	Recovery of spiked compound in sample matrix	Used to assess matrix effects and accuracy

Table 5-2	Summary of Formula Calculations
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5.2.2.6 Sensitivity

Sensitivity expresses the degree of response a given analytical method has to a given target analyte. It is usually defined in terms of an LOD or LOQ for a given matrix.

5.2.2.7 Limit of Detection

The Limit of Detection (LOD) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish LODs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these LODs at least once per 12-month period. Results less than or equal to the LOD shall be reported as the LOD value and flagged with a "U" (see Table 5-3).

Qualifier	Description		
J	The analyte was positively identified; the quantitation is an estimation.		
U	The analyte was analyzed for, but not detected at the LOD.		
R	The data are unusable because of deficiencies in the ability to analyze the samp and meet QC criteria.		
В	The analyte was detected in an associated blank at a concentration less than ten times the reported concentration in the sample.		
М	M A matrix effect was present.		
QH, QL	One or more QC criteria, such as surrogate or LCS recovery, failed with a high or low bias.		

Table 5-3 Data Qualifiers

Notes:

LCS	=	laboratory control sample
LOD	=	limit of detection
QC	=	quality control

5.2.2.8 Method Blank and Trip Blank Contamination

When positive results are reported in either the trip blank or method blank, sample results less than ten times the reported concentrations in the blanks will be qualified with a B to indicate potential high bias due to blank contamination. This rule applies to all results and not just those reported at concentrations greater than one-half the reporting limit.

5.2.2.9 Limit of Quantitation

The laboratory participating in this work effort shall compare the results of the LOD demonstrations to the LOQs for each method used on this project. The LOQ must be a minimum three times the LOD. The laboratories shall also verify LOQs by including a standard at, or below, the LOQ as the lowest point on the calibration curve. All results shall be reported at, or above, the LOD values; however, for those results falling between the LOD and the LOQ, a "J" flag shall be applied to the results indicating the variability associated with the result (see Table 5-2).

5.3 QUALITY CONTROL AND SAMPLING

5.3.1 Sampling Locations

The specific sampling locations are identified and discussed in the Work Plan.

5.3.2 Sampling Procedures and Protocols

Sections 3.0 and 4.0 of this document and information provided in the Work Plan detail sampling locations and sampling techniques, decontamination procedures, and sampling equipment. Calibration procedures are addressed in the specific manuals and SOPs found in Appendix F of this SAP. Specific QC and documentation protocols applicable to sampling procedures are discussed in Sections 4.6 and 4.8 of the SAP. The SOPs are based on acceptable State of Alaska, DoD, and EPA practices. Conventional sampling practices will be followed.

5.3.3 Sample Volume

The volumes of samples, containers, bottle types, and preservatives have been established by the USACE and are listed in Table 4-1 in Section 4.0, the Field Sampling Plan of this document.

5.3.4 Elements of Quality Control

This section presents QC requirements relevant to the analysis of environmental samples that shall be followed during all analytical activities at the project laboratory. Laboratory QC samples (e.g., blanks and laboratory control samples) shall be included in the preparation

batch with the field samples. A preparatory batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and that are extracted or digested at the same time, with the same lot of reagents. The MSs and MSDs count as QC samples. No MS/MSD samples will be extracted in the field laboratory.

Continuing calibration verification (CCV) standards are typically analyzed every 12 hours within an analytical sequence, with the exception of AK methods which require CCVs every 20 samples. The identity of each analytical batch shall be unambiguously reported with the analyses, so that a reviewer can identify the QC samples and the associated environmental samples. The type of QC samples and the frequency of use of these samples are discussed below.

Acceptance criteria and possible qualification (flagging) of analytical results will use appropriate USACE designations. Assignment of flags will be based on criteria set forth in the respective federal or state methods (EPA, 1986; ADEC, 2002) under EPA Solid Waste Method SW846 and Alaska methods, as described in the Underground Storage Tanks Procedures Manual.

5.3.4.1 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but are not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates shall be added to environmental samples, controls, and blanks, in accordance with the method requirements. Whenever a surrogate recovery is outside the acceptance limit, corrective action (Section 5.9) must be performed. After the system problems have been resolved and system control has been reestablished, reprepare and reanalyze the sample. If corrective actions are not performed or are ineffective, the appropriate data qualifier, as described in Table 5-2 and Section 5.9 shall be applied to the sample results.

5.3.4.2 Field Duplicates

For every 10 samples collected, one duplicate sample will be collected and submitted for laboratory analysis per each matrix. The duplicate sample is designed to be identical to the original sample and is submitted to gain precision information about homogeneity, handling, shipping, storage and preparation, and analysis. Duplicate sampling is used to identify possible field variations. The duplicate sample will be collected at the same time and location as the environmental sample. Waste characterization samples will also have field duplicate samples submitted for analyses.

5.3.4.3 Matrix Spike/Matrix Spike Duplicates

An MS/MSD is an aliquot of sample spiked with known concentrations of all target analytes. The spiking occurs before sample preparation and analysis. The MS/MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Only samples generated for this project shall be used for spiking. The MS/MSD shall be designated on the CoC form. The MS/MSD is used to document the bias of a method due to sample matrix.

A minimum of one MS and one MSD sample shall be analyzed for every 20 confirmation samples per matrix. The performance of the MS/MSD is evaluated against the LCS acceptance limits. When initial sample concentrations are greater than 4 times the spiked concentrations, MS/MSD recoveries are not evaluated. Waste characterization and field-screening samples do not require MS/MSD samples.

5.3.4.4 <u>Retention Time Window</u>

Retention time windows are used in GC and high-performance liquid chromatography analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are provided in SW846 Method 8000C. When the retention time is outside the acceptance limits, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze all samples analyzed since the last acceptable retention time check.

5.3.4.5 Method Blanks

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank shall be carried through the complete sample preparation and analytical procedure. It is used to document contamination resulting from the analytical process and shall be included in every analytical batch.

The presence of analytes in a method blank at concentrations greater than or equal to half of the LOQ and is greater than 1/10 the amount measured in any sample indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination before proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch shall be reprepared and reanalyzed. No analytical data shall be corrected for the presence of analytes in blanks. When analytes are detected in the method blank and samples at less than 10 times the concentrations are greater than 10 times the concentration reported in the method blank, no flags will be assigned to those analytes.

5.3.4.6 Holding Time Compliance

All sample preparation and analysis shall be completed within the method-required holding times. The holding time begins at the time of sample collection. Some methods have more than one holding time requirement, such as for preparation and analysis (e.g., Methods AK 102, SW8082, etc.). The preparation holding time is calculated from the time of sample collection to the time of initiation of the sample preparation process, as described in the applicable method, before any necessary extract cleanup and/or volume reduction procedures. If no preparation (e.g., extraction) is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation before analysis, the analysis holding time is calculated from the time of preparation completion to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. Holding times are specified in Table 4-1.

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5.3.4.7 Confirmation

Qualitative confirmation of PCB sample results at or above the LOD will be achieved using pattern recognition. PCBs as Aroclors have distinct patterns which are related to their manufacture. Historical results have indicated that only Aroclor 1254 and Aroclor 1260 have been identified at NE Cape. Prior to sample quantitation, the analyst will use experience and pattern recognition to correctly identify which Aroclor is being quantitated.

There is no confirmation for DRO, RRO, and metals.

5.3.4.8 Trip Blanks

Trip blanks are prepared by the confirmation laboratory and accompany glassware from the time the sample containers leave the laboratory until they return. They are not opened in the field. Trip blanks will be included in sample shipments and analyzed with volatile samples as part of the QA/QC protocol. Sample shipments that do not contain volatile samples (GRO, EPA 8260) will not include trip blanks.

5.4 SAMPLE PRESERVATION

It is important to maintain the integrity of the samples from the time they are collected until the analyses are completed. The samples shall, therefore, be preserved at the time of collection, before transportation and storage, to prevent or retard degradation or modification of chemicals in samples. Sample preservation requirements are described in Table 4-1, which involves keeping the sample between 2 and 6 degrees Celsius at all times until the sample is extracted. Temperature blanks will be included in all shipments to TestAmerica to assure that samples have been properly cooled during shipment.

5.5 SAMPLE CUSTODY AND HOLDING TIME REQUIREMENTS

The history of each sample and how the sample is handled will be documented from the time the sample is collected through all transfers of custody until it is received at the analytical laboratory. Internal laboratory records will then document the custody of the sample through final disposition. Detailed procedures for sample custody are included in Section 4.8. A sample is considered to be in someone's custody if:

- It is in one's actual physical possession;
- It is in one's view, after being in one's physical possession;
- It is in one's physical possession and then locked or otherwise sealed so that tampering would be evident; or
- It is kept in a secure area, restricted to authorized personnel only.

5.5.1 Laboratory Custody Procedures

The laboratory custody procedures are as follows:

- 1. A sample custodian or designated alternate accepts custody of the shipped samples and verifies that the information on the sample labels matches the information on the CoC forms. Pertinent information regarding shipment, pickup, courier, etc., is entered in the "Remarks" section. The custodian then enters the sample label data into the sample tracking system of the laboratory. This system will use the sample label number or assign a unique laboratory number to each sample label, and will ensure that all samples are transferred to the proper analyst and are stored in the appropriate secure area according to method specifications.
- 2. Samples are distributed to the appropriate analysts as described in laboratory procedures. Laboratory personnel are responsible for the care and custody of the samples from the time they are received until the time each sample is exhausted or dispersed. All samples and extracts will be held for a minimum of 30 days or until the end of the project, whichever is greater. Archived samples must be kept in a preserved state until released by the Project Manager or designee (typically the Project Chemist).
- 3. When sample analyses and necessary QA checks have been completed in the laboratory, the unused portion of each sample and the sample container must be properly disposed of in accordance with all federal and state laws, rules, and regulations. Sample and extract disposal will be the responsibility of the laboratory. All identifying tags, data sheets, CoC forms, and laboratory records will be retained as part of the permanent documentation. Samples received by the laboratory will be retained until analyses and QA checks are completed.

5.5.2 Holding Time Requirements

The holding time requirements will follow EPA or State method guidance. Table 4-1 summarizes these requirements. Because of the remote nature and limited accessibility of the NE Cape site, environmental samplers will consider holding time requirements before sample collection. However, uncontrollable events and unforeseen weather conditions may preclude compliance with all agency-recommended holding requirements.

5.5.3 Corrective Actions

The laboratory will maintain an SOP for sample log-in. Irregularities noted on the log-in form will be reported to the lead Environmental Sampler. Corrective actions for warm coolers and/or poor sample condition may include the flagging of associated analytical results or re-sampling, in accordance with the direction of the Project Chemist in consultation with the USACE. Data associated with samples received outside of standard holding times will be flagged in accordance with general EPA and associated USACE guidelines.

5.6 ANALYTICAL PROCEDURES

5.6.1 Background

The analytical methods, both qualitative and quantitative, implemented in the field and at the laboratory will comply with State of Alaska and EPA-approved guidelines. The project analytical laboratory will be TestAmerica located in Tacoma, Washington. TestAmerica has USACE certification for the applicable analytical protocols in accordance with EM 200-1-3 *Engineering and Design – Requirements for the Preparation of Sampling and Analysis Plans* (USACE, 2001) and the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM v 4.1, April 2009), and holds current National Environmental Laboratory Accreditation for all appropriate fields of testing. TestAmerica's Self-Declaration Form and Laboratory Certifications are included in Appendix B.

5.6.2 Specific Analytical Chemical Procedures

Standard EPA and ADEC methods are referenced in this SAP. Table 5-4 summarizes the analytical procedures and their respective preparation methods for use in this project. All soil results will be reported on a dry-weight basis.

Field screening measurements will be made following procedures described in Section 4.2. In general, this requires strict adherence to the equipment manufacturers' directions or specifications for operation, including all maintenance and calibration requirements.

Parameter			Analytical Method	an a	Preparatory Methods Soil			Preparatory Methods Waste
DRO			AK 102		SW3550B			SW3550B
RRO			AK 103		SW3550B			SW3550B
Metals			EPA 6020/6010B		SW3050			SW3050
Total Organic Carbon (TOC)			EPA 9060		NA			NA
PCBs			EPA 8082		SW3550B			SW3580A
Notes:								
AK	=	Alaska Method			PCBs	=	polychic	rinated biphenyls
DRO	=	diesel range organics			RRO	=	residual	range organics
EPA = U.S. Environmental F			Protection Agency		SW	=	EPA So	lid Waste Method
NA	=	not applicable						

Table 5-4 Analytical Procedures

Analytical method details, including the LOQ and acceptance criteria for laboratory QC samples are presented in Table 5-4 for soil. These tables do not include the analyses for waste characterization, but control limits for all analyses are provided in Appendix G, Laboratory Control Limits.

5.6.3 Test Methods

The methods for conducting the analyses will follow standard EPA and ADEC protocols. The methods are listed in Table 5-3. Field measurements will be performed following the SOPs presented in this SAP.

5.6.4 Control of Testing

The laboratory program for controlling the testing of project samples is described in TestAmerica's approved Laboratory QA Plan. Field measurements will follow the SOPs, as presented in this SAP.

5.6.5 Limits of Quantitation

All empirical analytical LOQs are less than cleanup levels stated in Tables A1, B1 and C in Section 341 of 18 AAC 75; which is the lowest of the ADEC pathway criteria for analytes of interest. Table 5-4 contains both the LOQ and pathway criteria values. The RPD control

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limits were provided by TestAmerica-Tacoma, Washington, which is the supporting laboratory providing the analyses.

5.6.6 Laboratory Practices

Analytical instruments shall be calibrated in accordance with the applicable analytical methods. All analytes reported shall be present in the initial and continuing calibrations. All results reported shall be within the calibration range. Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials. Instrument calibration shall be checked using all of the analytes listed in the QC acceptance criteria (Table 5-4). All calibration criteria shall satisfy EPA SW846 and ADEC method requirements at a minimum. The initial calibration shall be checked at the frequency specified in the method, using materials prepared independently of the calibration standards. Analyte concentrations are determined with either calibration curves or response factors (RFs). For GC methods, when using RFs to determine analyte concentrations, the average RF from the initial five-point calibration shall be used. The continuing calibration shall not be used to update the RFs from the initial five-point calibration.

5.6.7 Standard Materials

Standard materials, including second-source materials, used in calibration and to prepare samples shall be traceable to National Institute of Standards and Technology (NIST), EPA, American Association for Laboratory Accreditation (A2LA), or other equivalent approved source, if available. If NIST, EPA, or A2LA standard material is not available, the standard material proposed for use shall be current, and the following expiration policy shall be followed:

The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be

based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be discarded. The laboratory shall label standard and QC materials with expiration dates.

A second-source standard is used to independently confirm initial calibration. A secondsource standard is a standard purchased from a different vendor than the vendor supplying the material used in the initial calibration standards.

5.6.8 General Procedures

Field and laboratory test equipment that does not meet specified QA requirements will be recalibrated in accordance with method specifications and manufacturer requirements as specified in the SOPs. When field test equipment is found to be out of calibration, damaged, lost, or stolen, an evaluation will be made to ascertain the validity of previous measurements and the acceptability of these results since the last calibration check. If measurements are suspected to be inaccurate or invalid, the original measurements and testing will be repeated using properly calibrated equipment, or the associated previous data will be flagged as suspect. Suspect measurements will be listed in a nonconformance report or a deficiency notice, as applicable.

Test equipment consistently found to be out of calibration will be repaired or replaced, and inspection and test reports will include identification of the test equipment used to perform the inspection and/or tests. A corrective action report will be completed for any instrument found to be defective, inoperable, or faulty. This report will include the identification of the instrument, date/time of the test, description of the test or evaluation, corrective action taken, and name and initials of the responsible party. This information will be noted in the instrument logbook.

5.7 INTERNAL QUALITY CONTROL CHECKS

5.7.1 Quality Control Checks

The Laboratory QA Officer is responsible for planning, scheduling, and coordinating evaluations of the internal QC checks in accordance with approved laboratory procedures. The Laboratory QA Officer will be able to provide to the Project Manager, upon request, a satisfactory evaluation of the following:

- Possession and use of the latest approved Laboratory QA Plan, SOPs, standards, and/or project specific instruction(s);
- Conformance with appropriate plans, procedures, standards, and instructions;
- Thoroughness of the performance;
- Identification and completeness of documentation generated during performance, including:
 - Project number and/or name,
 - Task description,
 - Name of performer, and
 - Date(s) of performance;
- Page number and total number of pages, if more than one sheet;
- Consideration of all blank titled spaces of forms;
- Legible and reproducible presentation;
- Reasonable data entries, calculations, and results;
- Precise plots, charts, data summaries, and graphs, and clearly defined parameters;
- Proper approval, transcription, and reference of input data; and
- Analysis of performance evaluation (QA/QC) samples as appropriate.

5.7.2 Acceptance Criteria

The following acceptance criteria will be considered if pertinent to the specific activity:

- Appropriate forms, logs, or formats have been used;
- Equipment has been referenced and calibrated as required; and
- Equipment meets specifications.

Other acceptance criteria are incorporated into the technical procedures that describe the performance and documentation of a specific activity.

5.7.3 Acceptance Documentation

A verifier will indicate acceptance of all work performed, as well as the resultant documentation, by signing (or initialing) and dating the appropriate form or space provided.

Differences between the verifier and work performer will be discussed and resolved. If agreement cannot be reached, the differences will be brought to the attention of successionally higher management levels until resolution is achieved.

5.7.4 Check Frequency

Undocumented checks (surveillance) may be performed, as assigned, during the activity. A check of documentation will be performed at the completion of the task.

5.7.5 Documentation

The checking function is documented in compliance with the applicable procedures for the specific task performed and retained for record purposes until job completion.

5.7.6 Analytical Laboratory

The internal QC procedures will be described in the Laboratory QA/QC Plan, together with associated implementing SOPs. The laboratory QA manuals and SOPs must be provided, upon request, to Bristol's Project Chemist or Project Manager for review and approval for use on this project. The following items should be covered in these procedures and plans:

- MS/MSD,
- Replicates,
- Blanks (field, trip, method, reagent instrument, decontamination, and source water),
- Internal standards (ISs) and surrogates,
- Calibration and calibration verification,
- Control charts,
- · Standards and standard sources, and
- Reagents and gases.

5.7.7 Field Sampling

5.7.7.1 Corrective Action

The Environmental Samplers may occasionally be required to adjust the sampling program to accommodate site-specific needs. If it becomes necessary to modify field sampling as described in this SAP, corrective action will be taken to ensure proper and approved procedures are implemented. If samples have been collected, these samples may be discarded and new samples collected. If samples have been sent for analysis, the laboratory may be contacted to terminate analysis. All corrective actions will be documented and reported immediately to the QAR and Bristol's Project Chemist and Project Manager.

5.7.7.2 Contamination

If sample results indicate contamination of field or trip blanks (detections above the LOQ), sampling and analysis may be performed again for the associated target analytes. The Project Manager, in conjunction with the Project Chemist, will make this decision.

5.8 DATA QUALITY INDICATORS AND DATA REVIEW

5.8.1 Data Quality

Data quality may be defined as the totality of features and characteristics of data that bears on its ability to satisfy a given purpose. The characteristics of major importance are precision, accuracy, representativeness, completeness, and comparability.

5.8.2 Fieldwork

Field sampling consists of a single collection cycle in the field for subsequent chemical analysis in an analytical laboratory. There may be no opportunity to make routine assessments of accuracy, precision, or completeness in the course of the field sampling. The QA/QC samples, as described in the FSP, will be included to assess fieldwork.

5.8.3 Laboratory Analysis

Information regarding the precision, accuracy, and completeness of data is compiled by the laboratory. The DQO requirements are presented in Section 5.2. LOQs are provided in Table 5-4. The methods for making these precision, accuracy, and completeness assessments

will be prescribed in the approved QAPP or SOPs of the analytical laboratory. These procedures will specify the processing of blanks, replicates, and spikes. Surrogate standards are used with each sample analyzed by GC. Additionally, the laboratory will monitor its QC data to ensure that they are within the established control limits for the methods, as published by the EPA or state agency.

Data accuracy and precision will be assessed for each sample lot using samples and sample duplicates spiked at a known level. Completeness will be reported. The descriptive calculations are presented in Table 5-1.

5.8.4 Procedure Validation

When new laboratory analytical methods are developed, the data necessary to characterize the method must be submitted to the Project Chemist or Project Manager before implementation. These data will include the implementing SOPs and results from MDL-LOD studies, results of MS and MSD tests (for accuracy and precision specifications), and other information sufficient to develop appropriate DQOs (such as surrogate recoveries, known interferences, and instrument specifications).

5.8.5 Review of Data

Data will be reviewed by internal laboratory QC personnel before submission to Bristol. Calculations will follow standard statistical conventions and formulas, as presented in Table 5-1. Third-party data review will be performed by AECOM[®]. If it is determined that any data need to be flagged, the qualifiers in Table 5-2 will be used. The ADEC laboratory check sheets will be completed for each analytical data package (with the exception of the waste data) and will be compliant with ADEC and DoD QSM criteria.

Bristol will provide one copy of the analytical data package to the USACE within 60 days after receipt of the last sample at the primary laboratory. The analytical data package will be provided to the USACE Project Chemist, Mr. Mike Utley, in hard copy and as a pdf. The analytical data package will include the information necessary to demonstrate that the project's DQOs have been fulfilled. The following data will be available in the analytical data package:

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- Chromatograms and other raw data;
- Internal QC data, including tabular summaries correlating sample identifiers with internal QC data (blanks, spikes, etc.); and
- Chemistry data in accordance with the Alaska District Standard Electronic Data File Format (SEDD Stage 2A Version 5.0), and the Corps of Engineers Loading Tool Electronic Deliverable Format (COELT EDF Version 1.2a).

5.9 CORRECTIVE ACTION

Corrective action will be initiated when potential or existing laboratory conditions are identified that may adversely affect data quantity or quality. Events that may lead to corrective action include the following:

- Violation of established analytical controls;
- Performance, system, or QA audits; and
- Laboratory/field comparison studies.

Corrective action may take several forms, but the following steps usually are included:

- Check the calculations,
- Check the instrument for proper setup, and
- Reanalyze the control item, as appropriate.

The Project Manager, Project Chemist, Environmental Samplers, and contract laboratory analysts may be involved in the corrective action. The corrective action may be immediate or long-term. An immediate corrective action may be recalculating, reanalyzing, or repeating sample collection. Long-term corrective action may be identified through performance evaluation samples, standards, control charts, or other devices.

Corrective actions, if necessary, are to be completed at once. If acceptance criteria were not met and a corrective action was not successful, or corrective action was not performed, the appropriate data qualifiers (Table 5-2) should be applied. Requirements and procedures for documenting the need for corrective actions are described in this section.

5.9.1 Corrective Action Report

Problems requiring corrective action in the laboratory will be documented by the use of a corrective action report. The Laboratory QA Officer or any other laboratory member can initiate the corrective action request in the event that QC results exceed acceptability limits or upon identification of some other laboratory problem. Corrective actions can include reanalysis of the sample or samples affected, resampling and analysis, or a change in procedures, depending upon the severity of the problem.

5.9.2 Laboratory Quality Control Samples/Surrogates

If laboratory QC samples and/or surrogates are outside control limit criteria, as defined in Table 5-4, corrective action will be initiated. Corrective action will be consistent with the requirements of the DoD's QSM, EM 200-1-3 *Engineering and Design Requirements for the Preparation of Sampling and Analysis Plans* (USACE, 2001), and the Laboratory QA Plan. This may take the form of re-extraction and reanalysis of the analytical batch. These data will be reviewed by the Project Chemist and, based on professional judgment, the data will be determined to be usable or not usable for intended purposes. If judged not usable, the Project Chemist will notify the Project Manager, and the decision for resampling/reanalysis will be determined on a case-by-case basis, depending on the needs for and uses of the particular data sets in question.

5.9.3 Performance and Systems Audits

If the performance or system audits identify deficiencies, these deficiencies will be documented in the audit report. In addition, a recommended list of corrective action items will be developed, specific to the auditor's findings, observations, and comments. The project technical staff will be solicited for input, as required, depending on the nature and extent of the findings. A copy of the audit report will be provided to the Project Manager. These items, depending upon the level of deficiency, will require follow up by the responsible parties and will be approved and closed by the auditor and Project Manager.

5.10 DATA REDUCTION, REPORTING, AND RECORDKEEPING

5.10.1 Data Reduction

5.10.1.1 Definition

The data generated for this investigation will be used to support site waste characterization, segregation, and remedial activities in a qualitative and, where appropriate, quantitative manner using a judgment-based approach.

5.10.1.2 Data Usage

The data generated at the site and/or in the laboratory will be used to support the professional judgment-based decisions and the activities described in the Work Plan. Results obtained from the field and confirmation laboratory is intended to be used for making characterization decisions regarding residual contamination in the field, as well as determining whether potential cleanup levels are achieved.

5.10.1.3 Data Reporting

All analytical and QC results will be verified with electronic error-free checker software against criteria specified in the project instructional set provided by the Alaska District, USACE chemist. Once the designated data review chemist reconciles discrepancies and resolves issues between the non-conformance report generated by the laboratory and the non-conformance report generated by the data review chemist, the electronic data deliverable (EDD) will be generated and released for incorporation into the final report. The final report issued by the laboratory will be the standard USACE report package. The EDDs will be in COELT 1.2a format, as well as USACE Staged Electronic Data Deliverable (SEDD) version 5.0 Stage 2A. The EDD laboratory data packages shall match the hard copy data deliverable supplied by the laboratories, including data qualifiers (flags).

The final report will include a Chemical Data Final Report (CDFR) and it will contain a summary of QC practices, and all chemical parameter measurement activities. The CDFR will include:

- Laboratory data;
- ADEC Laboratory Data Review Checklist (Appendix H);

- Summary of the project scope and description;
- Summary of any deviations from the design chemical parameter measurement specifications;
- Summary of chemical parameter measurements performed as contingent measurements;
- Summary discussion of resulting data, including achieving data reporting requirements;
- Summary of achieving project-specific DQOs;
- Summary of field and laboratory oversight activities, providing a discussion of the reliability of data, QC problems, and a summary of the evaluation of data; and
- Conclusions and recommendations.

5.10.1.4 Supplementary Data

Supplementary data produced for internal records and not reported as part of the analytical data may include laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standards records, maintenance records, calibration records, and associated QC records. These sources will be available for inspection during audits and for use to determine the validity of data.

Data from other sources will not be used in project analysis or reports until the laboratory Project Chemist can be assured that the data were collected and analyzed according to procedures and protocols specified in this QAPP and associated FSP (Section 4.0). The source of outside data will be included in project reports in which these data are used.

5.10.2 Recordkeeping

The laboratory shall maintain electronic and hard-copy records sufficient to recreate each analytical event conducted pursuant to the Statement of Work for a minimum of five years. The minimum records the laboratory shall keep include the following:

- CoC forms;
- Initial and continuing calibration records, including standards preparation, traceable to the original material and lot number;
- Instrument tuning records (as applicable);
- Method blank results;

- IS results;
- Surrogate spiking records and results (as applicable);
- Spike and spike duplicate records and results;
- Laboratory records;
- Raw data, including instrument printouts, benchwork sheets, and/or chromatograms with compound identification and quantitation reports;
- Corrective action reports;
- Other method- and project-required QC samples and results; and
- Laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

5.10.3 Correction to Documents

If an error is made during data reduction, analysis, or reporting, the error will be corrected by lining through the error so as not to obscure the original entry, then entering the correct information, and initialing and dating the entry.

5.11 PREVENTATIVE MAINTENANCE

The objective of the preventive maintenance program for sampling and analytical equipment is to avoid generating spurious environmental measurements that could lead to inappropriate remedial responses. The preventive maintenance program is described in detail in the FSP.

5.11.1 Sampling and Analytical Equipment

Field sampling and analytical equipment affecting project data will be kept in good working order. Records of equipment maintenance will be maintained in Bristol's Anchorage office. If leased, maintenance records must be kept by the vendor and made available upon request.

5.11.2 Laboratory Preventive Maintenance

Laboratory preventive maintenance will be implemented in accordance with the Laboratory QA Plan and associated implementing SOPs. At a minimum, all major instrumentation will have associated records and logbooks, including schedules and criteria for maintenance.

5.12 PERFORMANCE/SYSTEMS AUDITS

5.12.1 Systems Audits

A systems audit of the analytical laboratory, field testing activities, and QC program will be conducted, if deemed necessary by the USACE, the Project Manager, or the Project Chemist. The systems audit will focus on the acceptability of project organization, staff, facilities, equipment, and methods.

The audit will cover, in general, verification that approved procedures, a calibration program, and an organization structure are in place and are used. The audit also will ensure that personnel responsibilities are clearly defined; a training program for personnel, CoC program, and records retention program are in place and are current; and corrective action of variances taken by laboratory and field personnel is responsive and timely. The systems audit will be conducted under the direction of the Project Manager and/or Project Chemist, their staff members, or a third-party agreed to by Bristol and the USACE.

5.12.1.1 Analytical Laboratories

Internal systems audits will be performed by the Laboratory QA Officer, as described in the laboratory's SOP Manual. Systems audits will involve laboratory comparison of project performance (as documented by protocols and procedures) to validate data. Results of the audits will be retained as a project record and made available to the Project Manager and/or Project Chemist on request, for use during subsequent laboratory systems audits.

Audit reports will be sent to the Project Manager and will be retained as a project record.

5.12.2 Periodic Surveillance by Laboratories

Laboratory activities that are subject to periodic review by internal laboratory QC personnel include the following:

- Review and approval of the Laboratory QA Plan;
- Review of parameter and/or laboratory notebooks;
- Review of instrument logs;
- Sample log-in, dispensing, and labeling for analysis;

Sampling and Analysis Plan Contract No. W911KB-10-C-0002

- Updating of QC criteria for spike recoveries;
- Final approval of data from each sample lot (field group); and
- Control of chemicals with limited shelf life.

These periodic surveillance activities will be conducted as described in the Laboratory QA Plan.

5.12.3 Performance Audits

Performance audits evaluate the actual performance of a laboratory. Audits are conducted periodically to determine the accuracy of the total measurement system(s) or parts thereof, typically against known Performance Evaluation (PE) standards. These standards can be blind PEs, provided by Bristol or an external third party, or known ISs, such as surrogates or MSs. Blind PEs will be submitted at the discretion of the Project Chemist. The source of these PEs may include NIST or other third-party vendors.

5.12.4 Resolution of Discrepancies

If there are any discrepancies, deficiencies, or indeterminate results in the field or laboratory, the individual who discovers the problem will take the appropriate corrective actions. If resolution cannot be reached immediately, the individual will bring the problem to the attention of the Project Manager or the Project Chemist to initiate resolution. If the problem cannot be rectified to the satisfaction of all concerned, the work will be stopped by the Project Manager until the situation is resolved.

The Project Manager will evaluate the problem, provide direction, and verify implementation of solutions before allowing the activity to resume. If appropriate, the laboratory will take the following actions:

- Bench Technicians will verify that the Laboratory Information Management System's output is correct, and follow SOP if the output is found to be out of compliance;
- Laboratory supervisors (or equivalent) will review all preliminary reports and submit any discrepancies to the Bench Technicians for review and possible corrections; and
- The Project Chemist will review all preliminary and final reports and, if obvious errors or discrepancies are identified, the Project Chemist will contact the laboratory and direct corrective actions.

5.13 QUALITY CONTROL REPORTS TO MANAGEMENT

At a minimum, the Project Manager shall prepare a summary report of the status of the project, QA/QC problems, corrective actions taken, and unresolved Recommendations for Corrective Actions with recommended solutions for management. The report shall also include results from all samples, the ADEC Laboratory Data Review Checklist (Appendix H), audit findings, and periodic data quality assessments.

6.0 REFERENCES

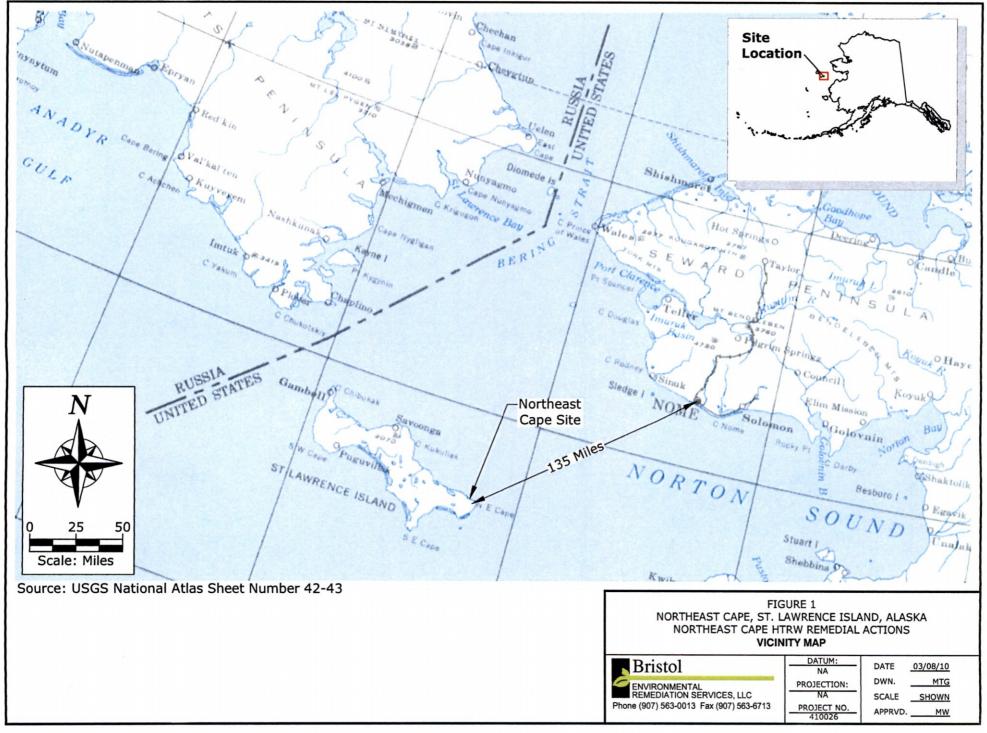
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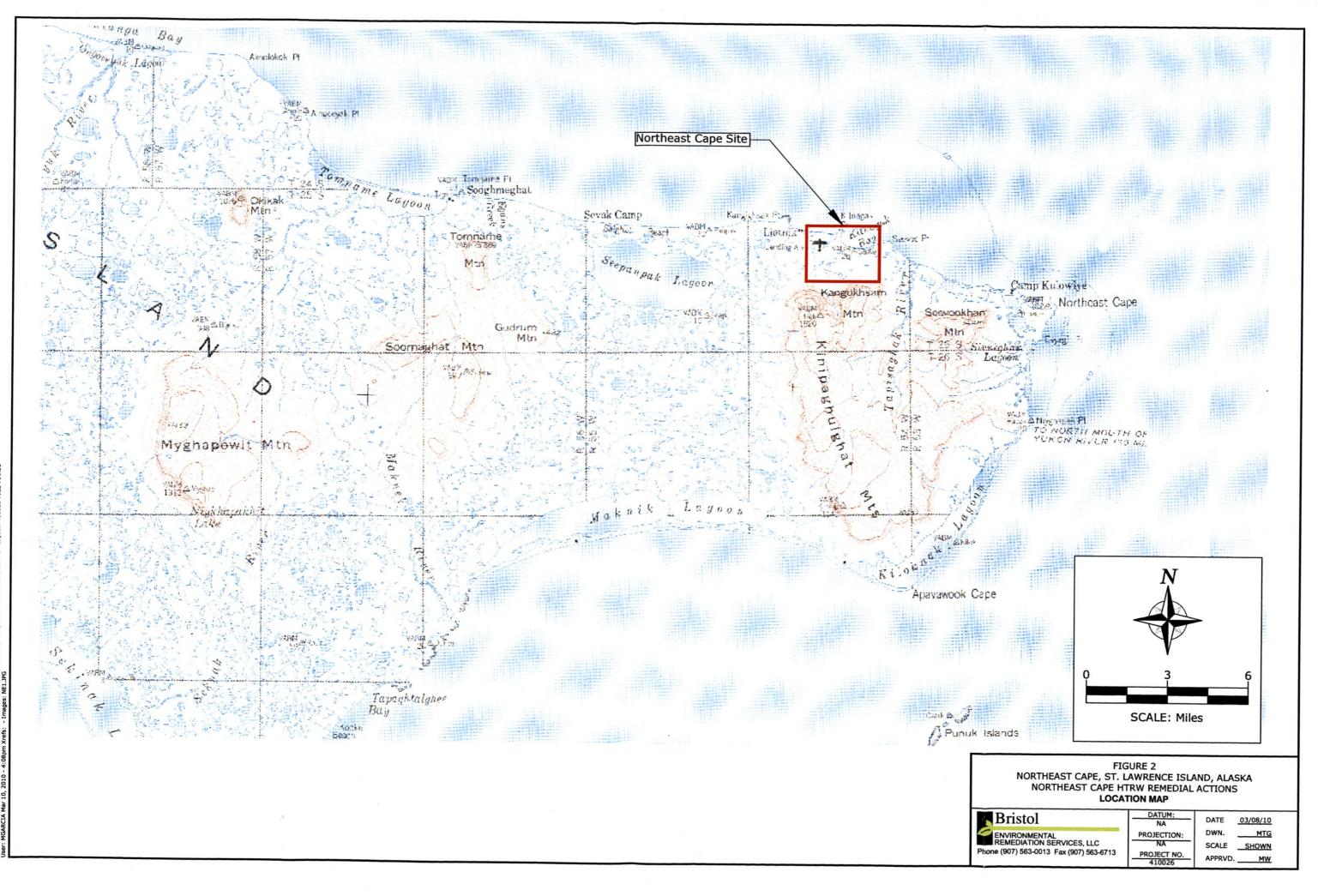
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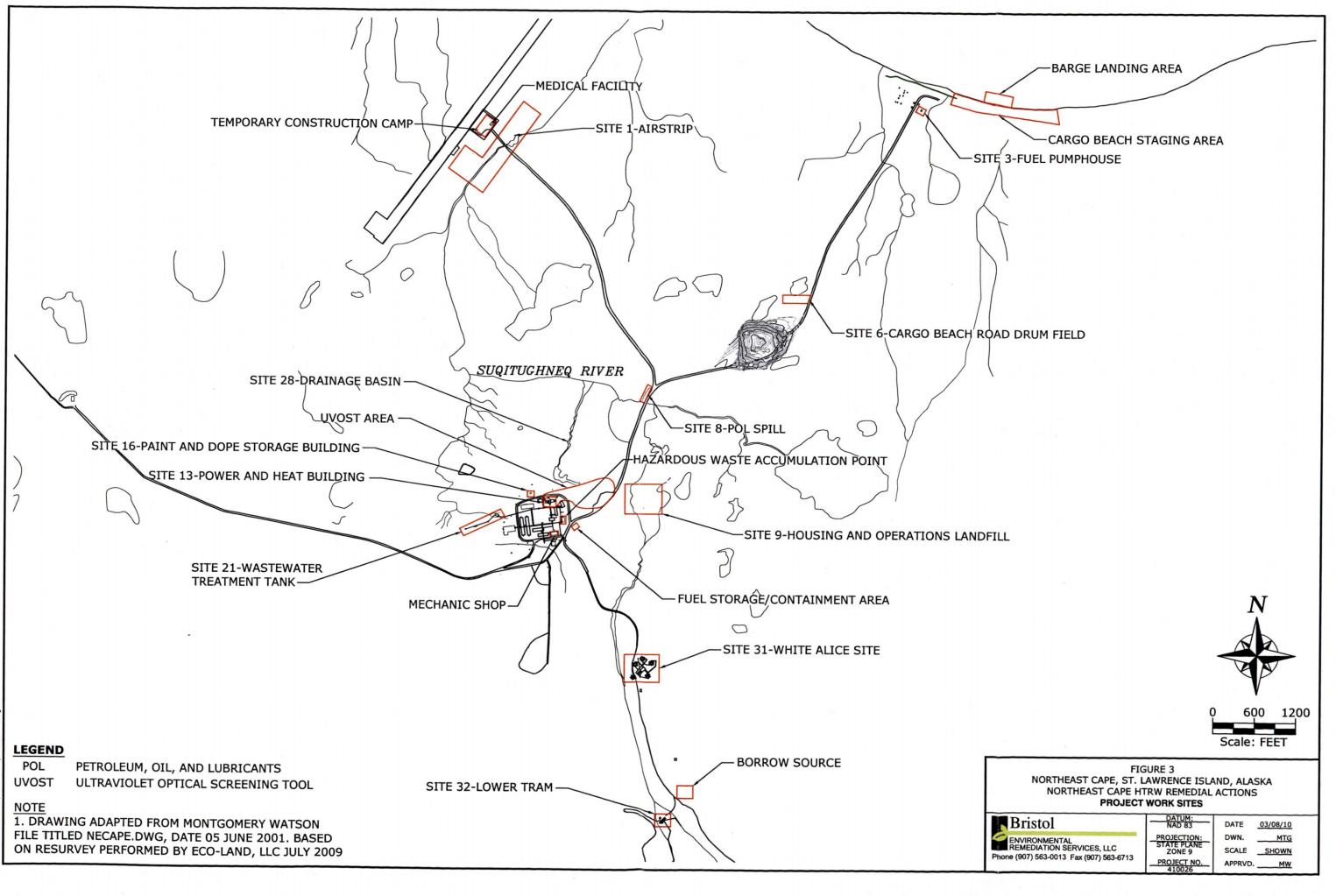
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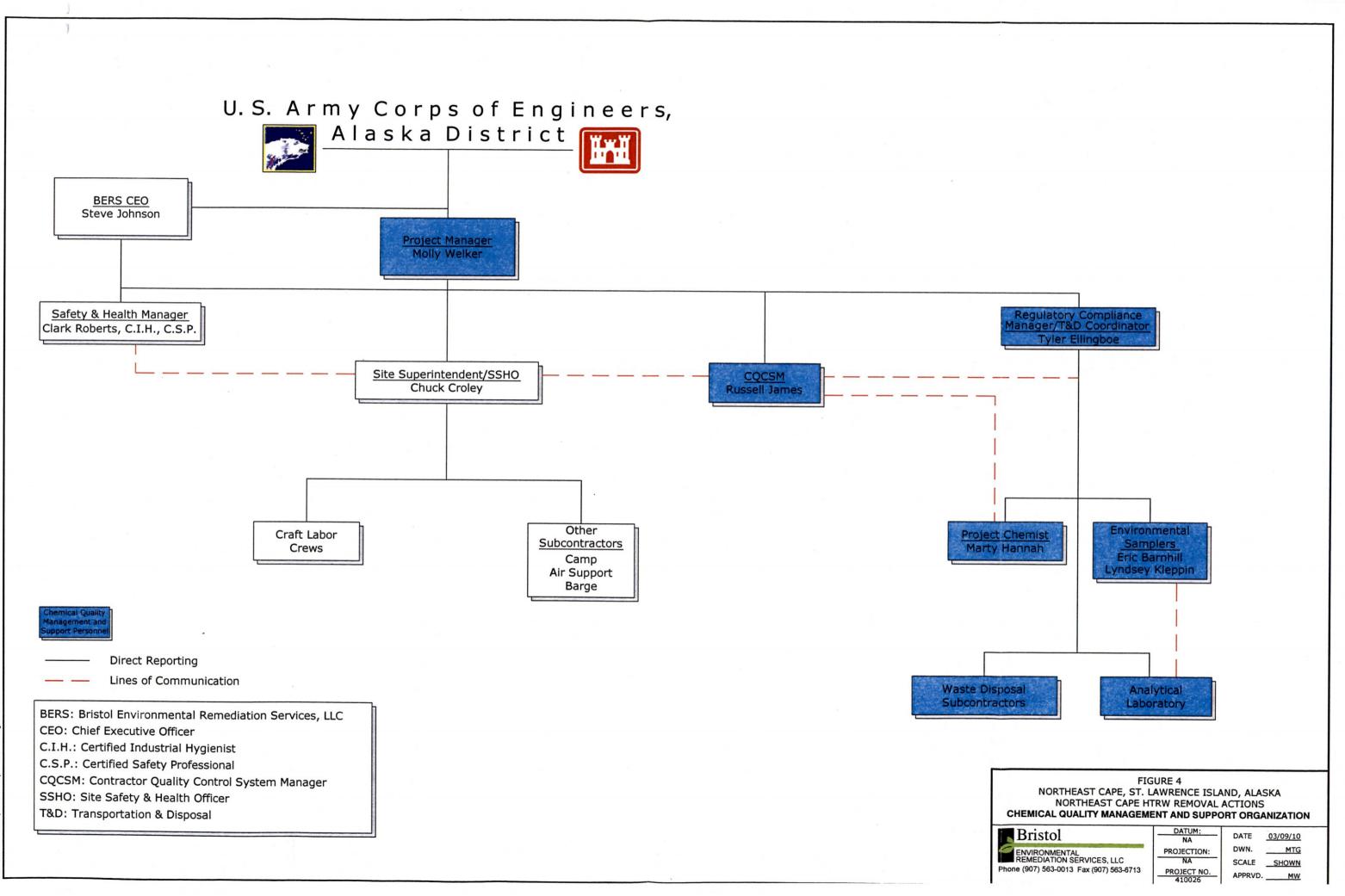


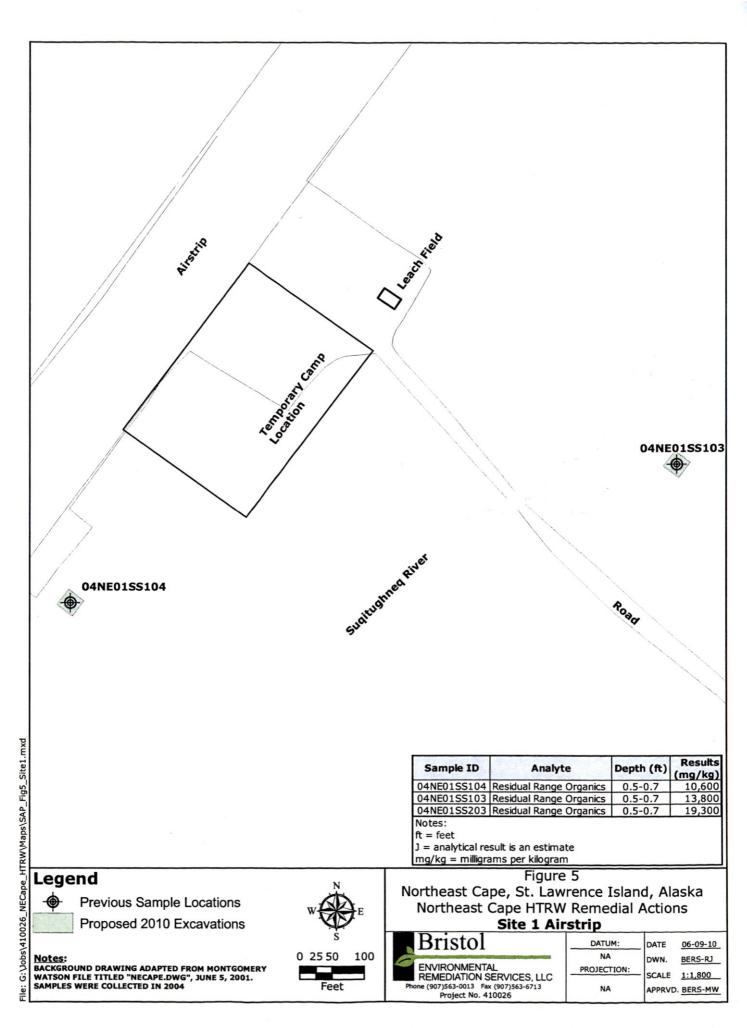


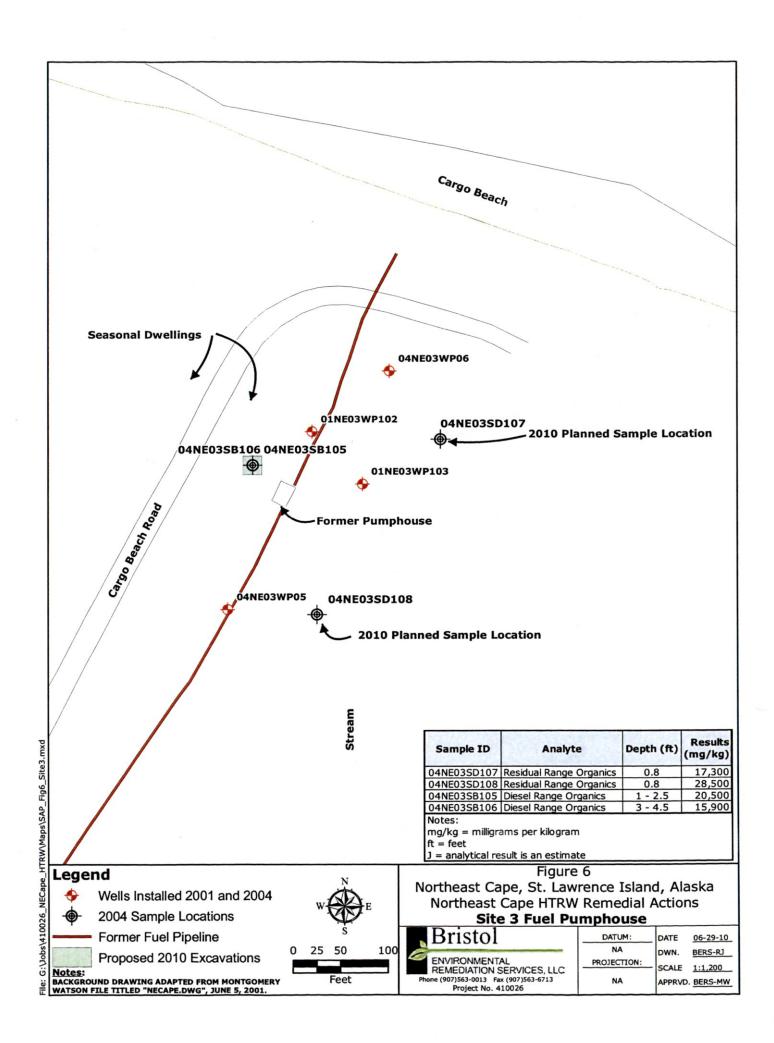
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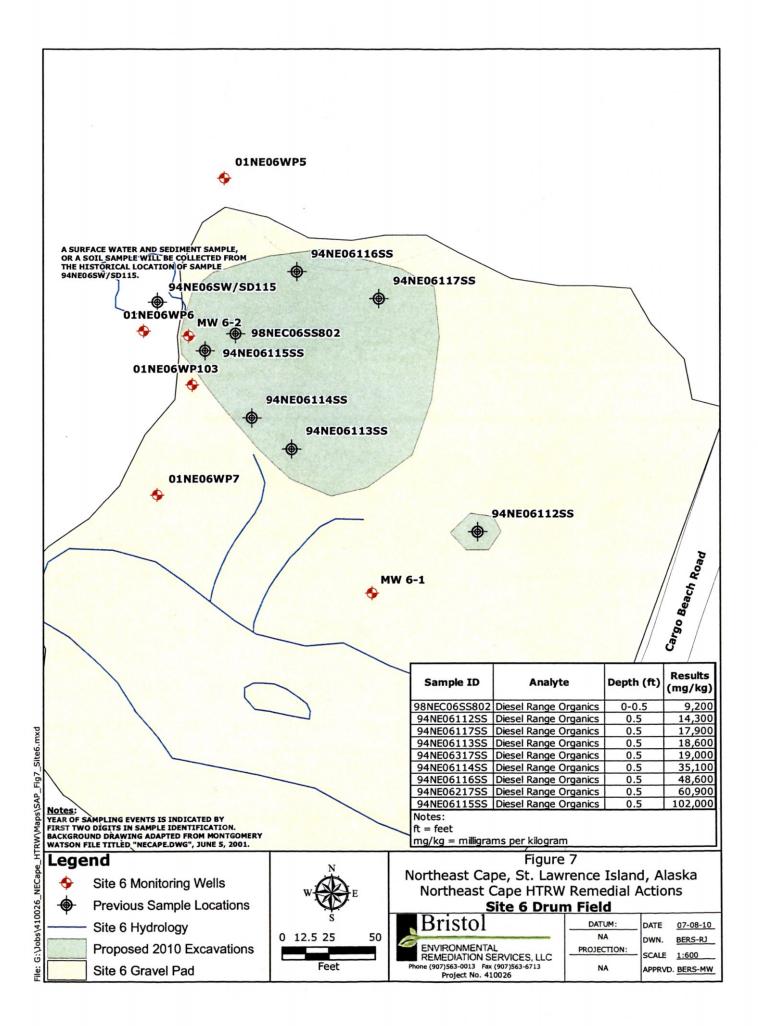


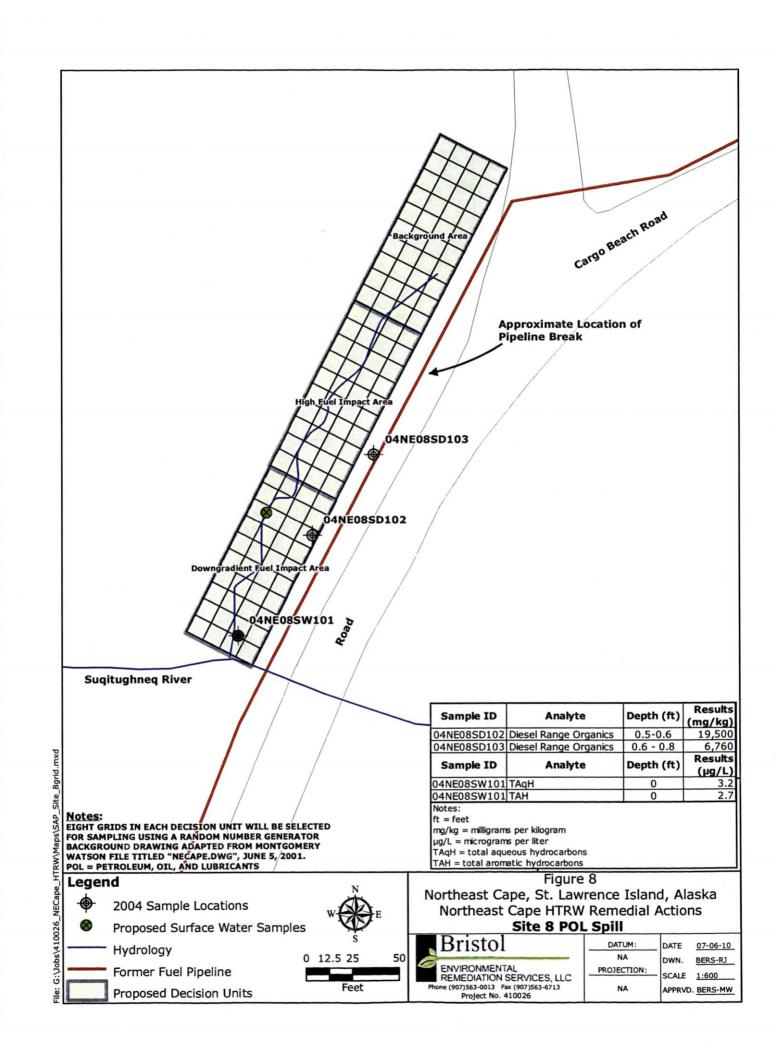
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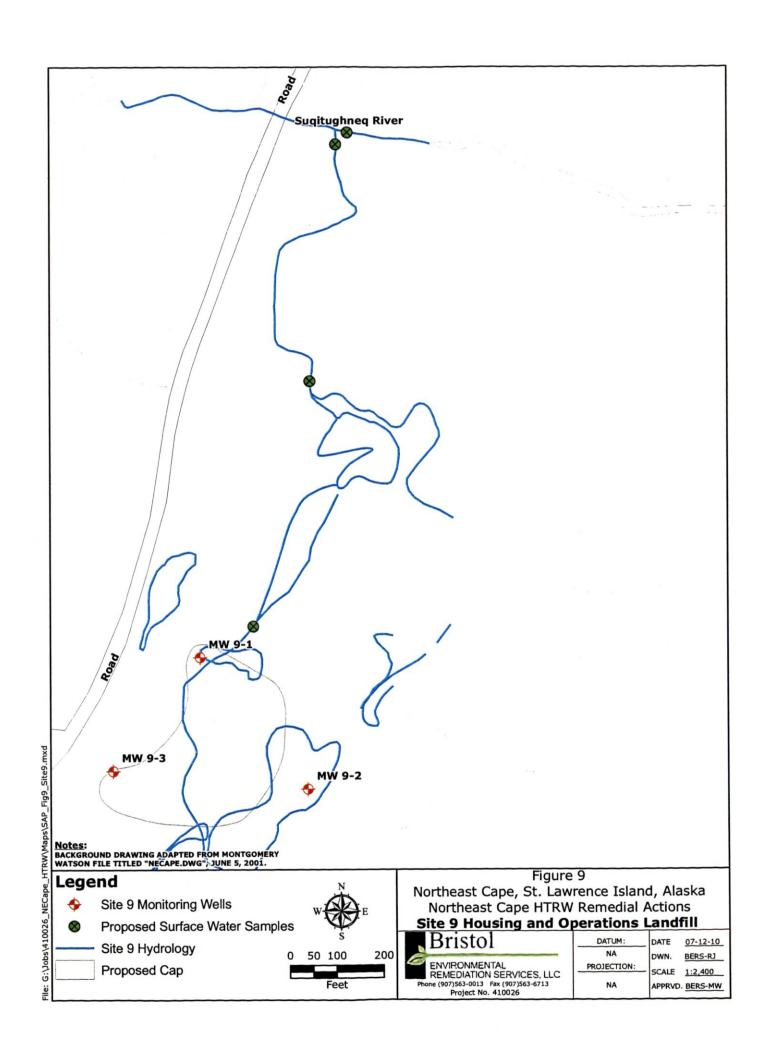


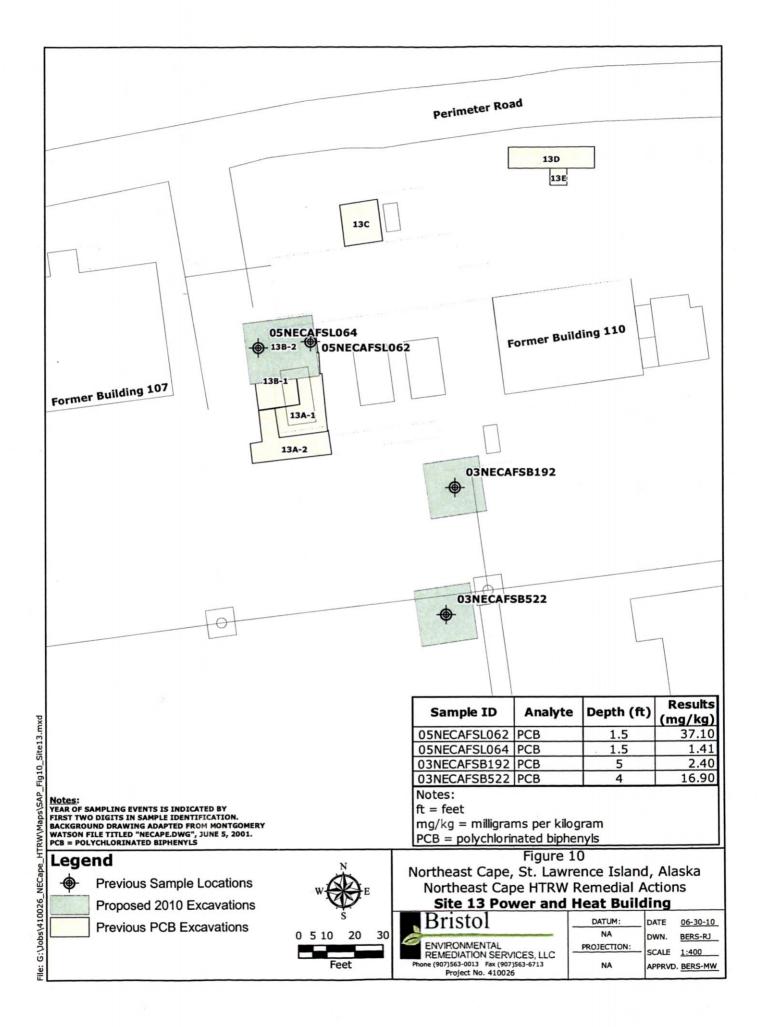


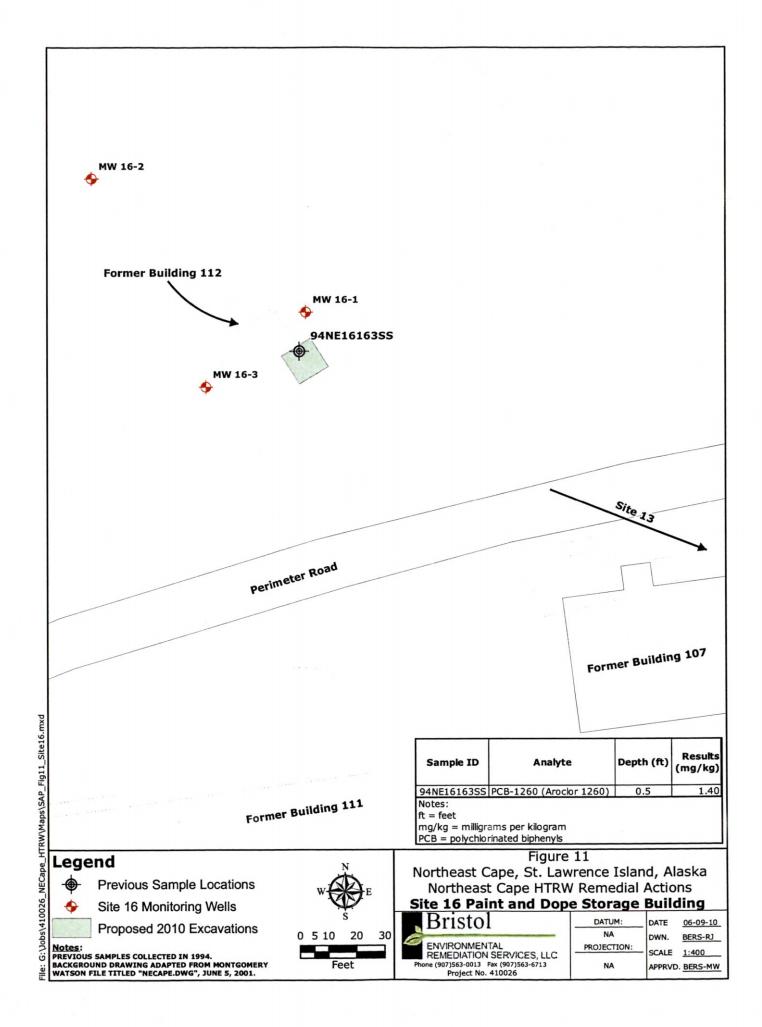




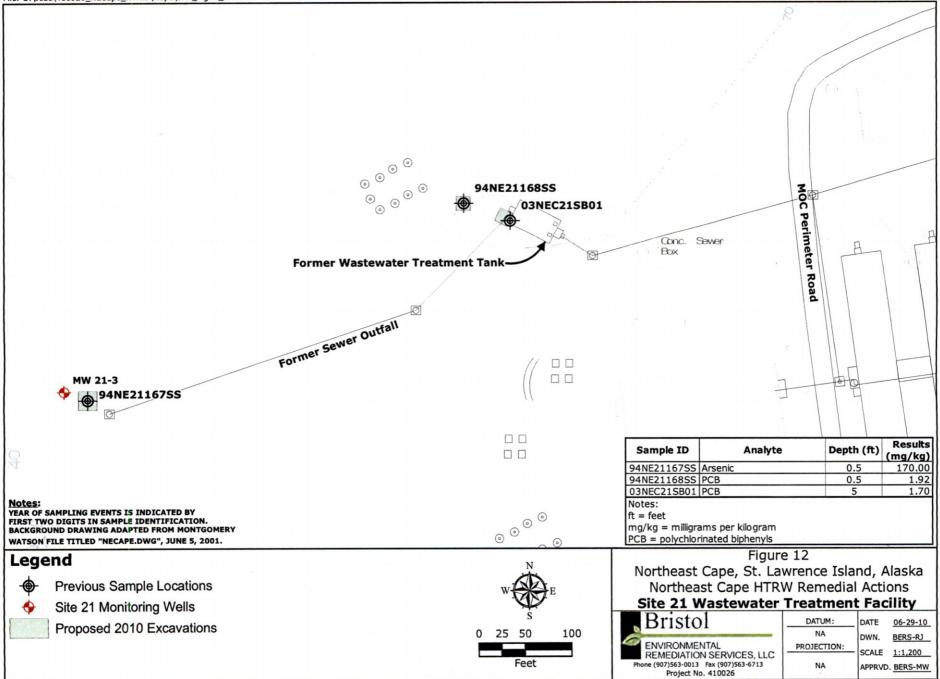


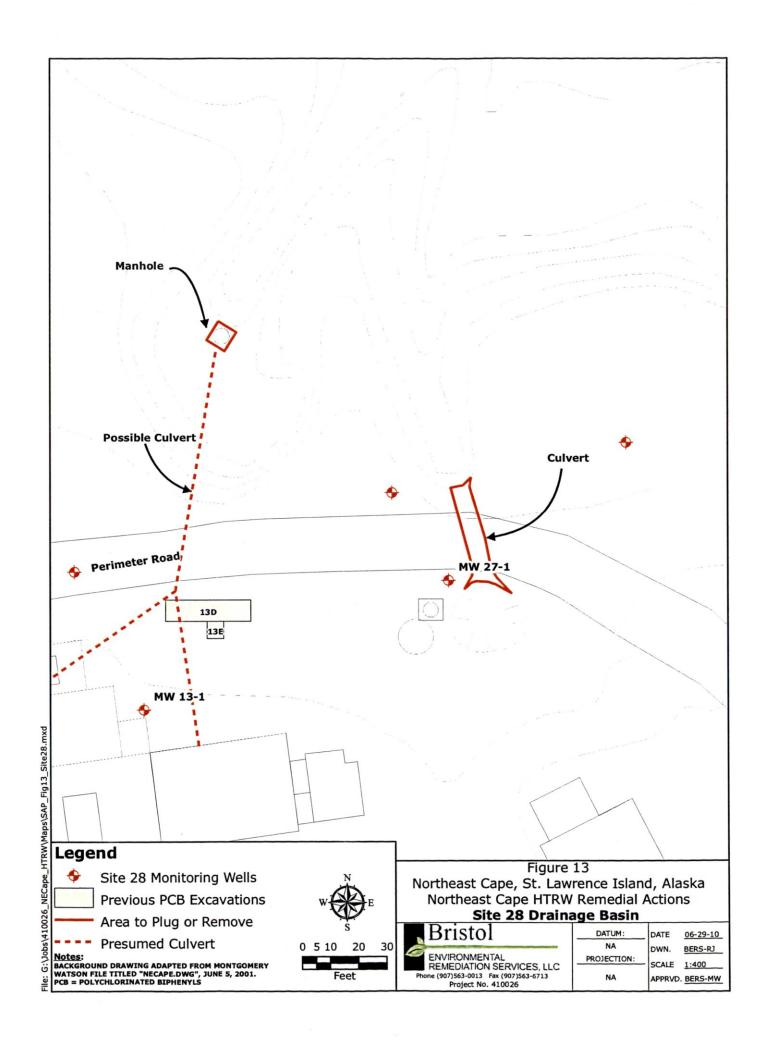


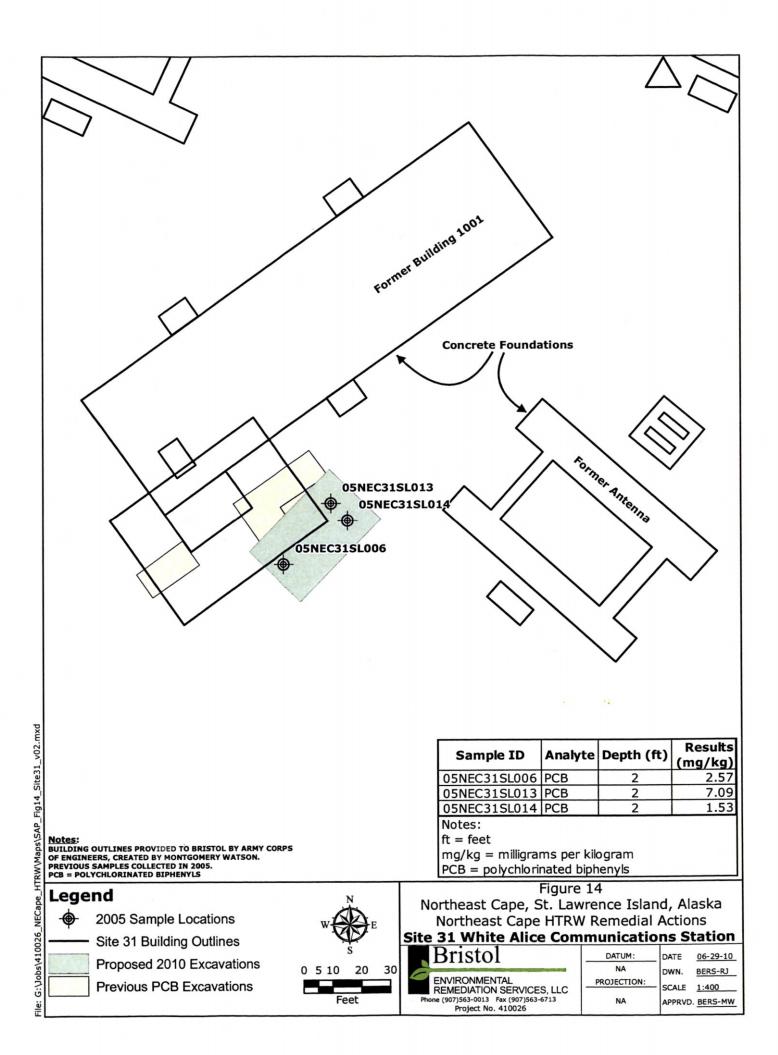


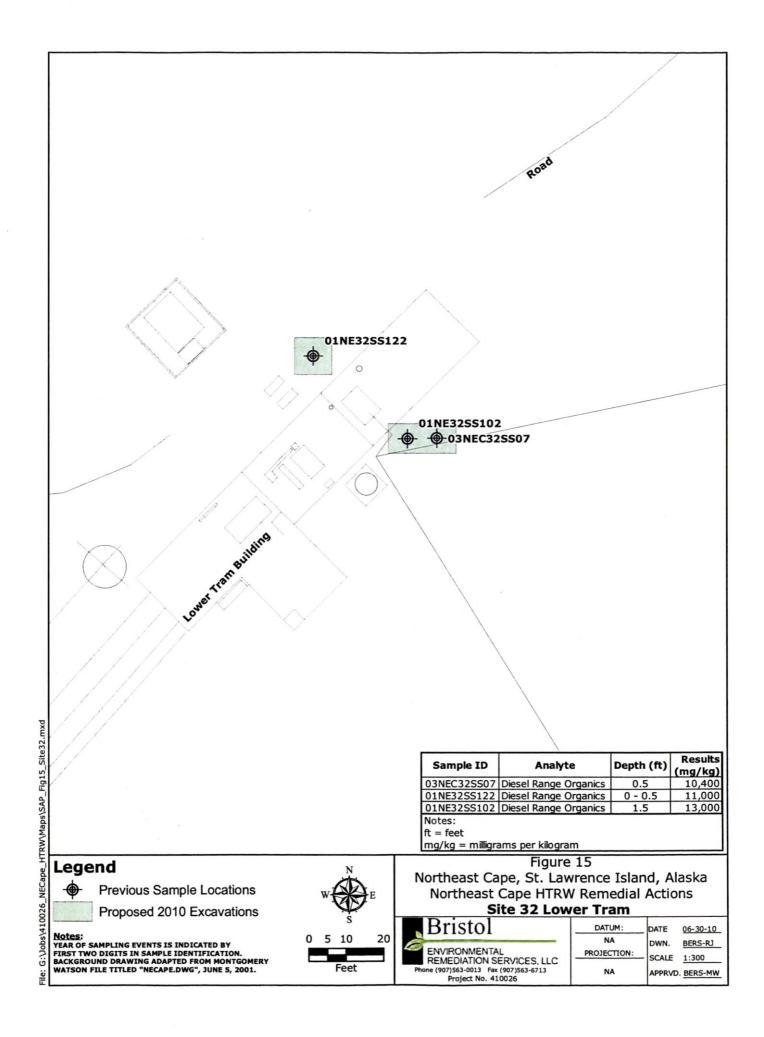


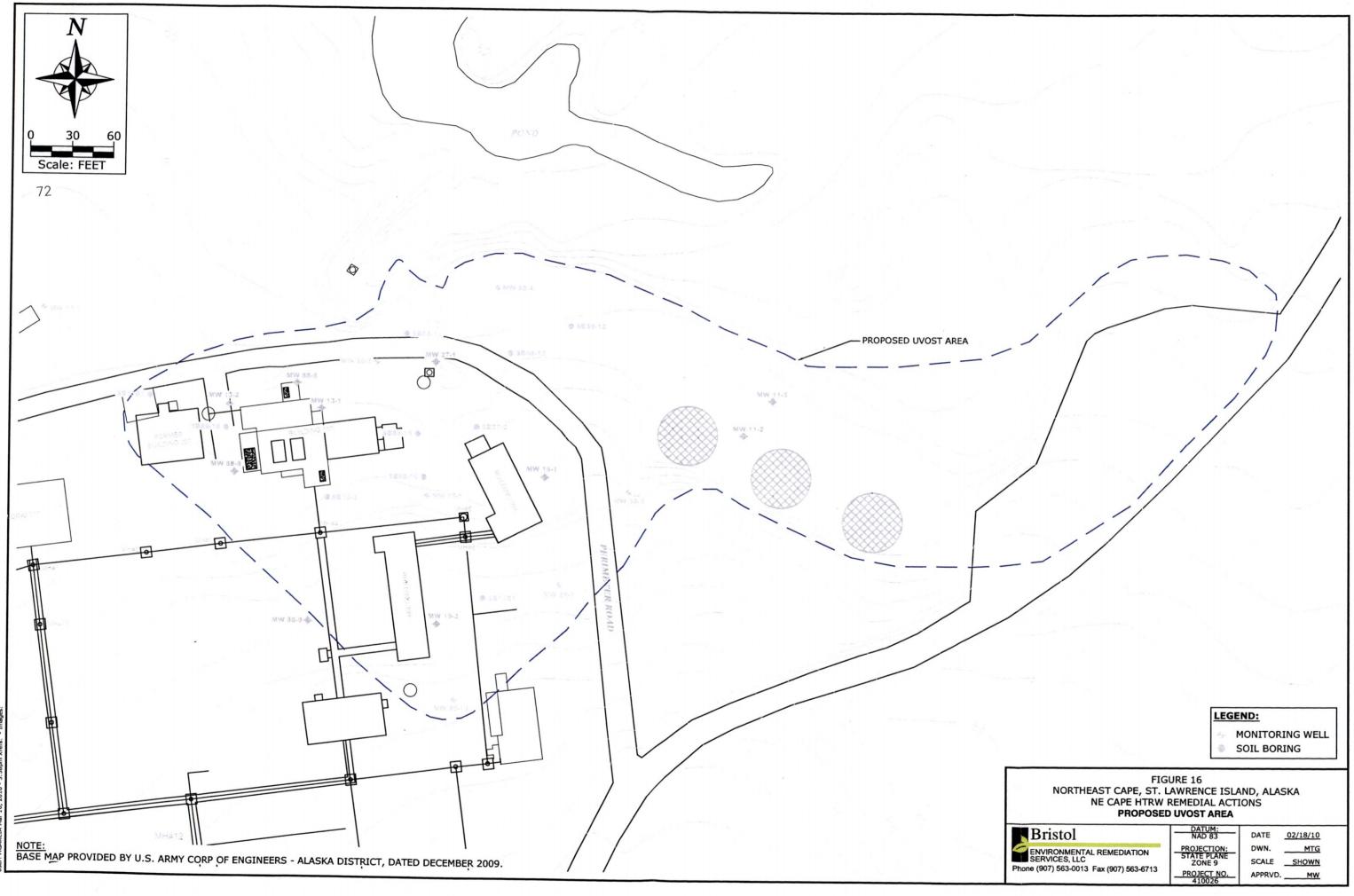
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APPENDIX A

Resumes

Molly Welker Charles Croley Russell James Marty Hannah Eric Barnhill Lyndsey Kleppin

MOLLY WELKER

Senior Project Manager

Areas of Expertise

- ✓ Project Management
- ✓ Regulatory Compliance
- ✓ Hydrology
- ✓ Geology
- ✓ Research
- ✓ Water, Stormwater, and Wastewater Analysis
- ✓ Water Quality Programs
- ✓ Environmental Baseline Studies
- Public Outreach and Education

Ms. Welker has developed and administered water quality monitoring programs for private, state, and federal agencies for more than 20 years. Her project responsibilities have included oversight of water monitoring programs, research, grant preparation and budget administration, public presentations, preparation of environmental baseline documents, and writing and editing. Ms. Welker is experienced in partnering with federal, state, city, and county government agencies, educational institutions, and environmental consulting firms. She serves on the board of directors for the non-profit Anchorage Waterways Council. Ms. Welker is well versed in regulatory compliance for Alaska Department of Conservation, U.S. Environmental Protection Agency (EPA), EPA Drinking Water Standards, Chronic Aquatic Life Criteria, National Pollutant Discharge Elimination System permits, the Total Maximum Daily Load Program, and Stormwater Pollution Prevention Plans.

Education

Master of Science, Geology, Texas A & M University, 1985

Bachelor of Science, Geology, Montana State University, Bozeman, 1982

Project Experience

- Senior Project Manager for Bristol Environmental Remediation Services, LLC, December 2006 to present.
 - Hoonah RRS Remedial Action Project Phase I and II (2006 to present). The project includes mobilization and demobilization to the site; excavation, sampling, and backfilling; transport of contaminated soils off site; and a final report. The work consists of excavation of approximately 1,271 tons of PCB-contaminated soil from a former soil stain area; a composite building area and generator room trench discharge area; a 32,000-gallon diesel underground storage tank (UST) area and radio relay building area; a gasoline UST and AST area and stockpile area; a bulk tank dike and fuel transfer area; and a former septic tank outfall. The former Hoonah RRS is a Formerly Used Defense Site (FUDS) located in southeast Alaska within the Tongass National Forest. It was a tropospheric station for Ballistic Missile Early Warning System and part of the White Alice Communication System.
 - Nuvagapak/Kogru/Collinson Remedial Investigation Project (2007). The project included preparing planning documents, mobilization and demobilization to and from the sites, performing a coastal erosion study and sample collection, sample transport and laboratory analysis, and final RI report for the Nuvagapak Point (BAR-A) Distant Early Warning (DEW) Line Station, Collinson Point (POW-D) DEW Line Station, and Kogru DEW Line Station. The sites are located on the remote northeast coast of Alaska within the Arctic Wildlife Refuge. The work included soil, water, and sediment sample

collection and analysis for various contaminants, including DRO, GRO, RRO, PCBs, PAH, TAHs, TAqHs, lead, and arsenic, from various areas of concern.

- Senior Project Manager for Bristol Environmental & Engineering Services Corporation, July 2006 to December 2006.
 - Northern Dynasty Mines, Inc., Illiamna area, Alaska. Performed project management activities and managed field program related to the Pebble Gold/Copper Mining project. Managed a team of interdisciplinary scientists conducting baseline field studies related to surface water quality, seep, and fine-grain bed sediment sampling in the project area.
- Project Manager/Lead Scientist for HDR Alaska, Inc., 2004 to 2006.
 - Northern Dynasty Mines, Inc., Illiamna area, Alaska. Responsibilities same as above.
 - Project Manager for Matanuska-Susitna Borough project, Alaska. Performed stormwater and wastewater analysis.
- Contract Technical Writer for U.S. Forest Service, Rocky Mountain Region, Denver, Colorado, 2002 to 2004. Editor and technical writer for a multiple-scale ecosystem assessment and conservation project, involving terrestrial, riparian, wetland, and aquatic ecosystems for the Species Conservation Project for Region 2 of the USDA Forest Service Water Outreach Coordinator for the City of Laramie, Wyoming, 1999 to 2002. Interfaced science, management, and public opinion for the protection and conservation of the City's drinking water supply. Served as editor and project manager of Laramie Regional Drinking Water Protection Plan. Managed state and federal grants, and wrote draft municipal ordinances, and quarterly and final reports. Responsible for budget administration and student intern supervision. Provided staff support to City Manager, City Council, and City/County Environmental Advisory Committee.
- Research Associate for the University of Wyoming, Water Resources Center, Laramie, Wyoming, 1997 to 1999. Successfully acquired state funding and drafted a statewide ambient groundwater quality monitoring plan for the Wyoming Department of Environmental Quality – Water Quality Division.
- Project Manager for Colorado State University, National Atmospheric Deposition Program, Fort Collins, Colorado, 1993 to 1997. Developed and administered an environmental monitoring program that collected weekly samples for total mercury in precipitation from more than 30 sites in the country. Responsibilities included fundraising, grant writing, budget administration, laboratory contract oversight, marketing, oral and written presentations, and development of field methodology, data collection, and quality assurance protocols.
- Associate Engineering Geologist for the California Department of Health Services, Toxic Waste Division, Sacramento, California, 1990. Provided technical reports for the cleanup and abatement of hazardous and toxic wastes at contaminated sites throughout the state. Reviewed geologic, engineering, and chemical data for proposed remedial actions. Interpreted state and federal water laws.
- Hydrologist for the U.S. Geological Survey (USGS), Water Resources Division, Sacramento, California, 1985 to1990. Assembled geohydrologic and geochemical information for analysis related to a regional surface and groundwater study. Supervised hydrologic technicians, performed quality assurance/quality control procedures, and published study results as USGS Water Supply Paper.

Molly Welker

Software Capabilities

Microsoft Suite Global Positioning Systems

Training and Certifications

First Aid/CPR, 2008

Hazardous Materials Transportation – (DOT/IATA) Section 1.5 IATA Compliance, February 2008

- Hazardous Materials Transportation (DOT/IATA) 49 CFR 172.700-704 Compliance, February 2008
- 8-Hour Hazardous Waste Operations & Emergency Response (HAZWOPER) Supervisor Training, 2008

8-Hour HAZWOPER Refresher Training, 2008

RCRA Hazardous Waste for Supervisors, 2008

Security Awareness Training, 2008

Army Corps of Engineers Construction Quality Management for Contractors, 2007-2012

40-Hour Hazardous Waste Operations & Emergency Response, 2007

Sampling for Defensible Environmental Decisions, 2006

Environmental Monitoring Workshop, 2005

Bear Safety, 2005

Hypothermia Awareness, 2005

Helicopter Safety, 2005

Remote Site Safety, 2005

Water and Boating Safety, 2005

Avalanche Awareness, 2005

Hazard Communication, 2004

Office Safety, 2004

Awards

USFS Certificate of Merit 2003

CHARLES L. (CHUCK) CROLEY

Senior Technician

Areas of Expertise

- ✓ Quality Control
- ✓ Site Superintendent
- ✓ Safety and Health Management
- ✓ Fuel Storage Tank (FST) Installation and Removal
- ✓ Well Drilling and Sampling
- Mobilization and Demobilization

Mr. Croley has worked on remote site projects throughout Alaska for more than 35 years. From 1968 to 1979, Mr. Croley worked for a variety of construction and drilling contractors that conducted soils and mining work. The soils investigations included work for geotechnical studies for the pre-Trans-Alaska oil pipeline. Projects in mining fields included mineral exploration and hydrological studies for dam foundations. Mr. Croley is an experienced Site Superintendent, Health and Safety Officer, and Contractor Quality Control Systems Manager (CQCSM) for projects encompassing construction, aboveground and belowground fuel tank installations and removals, monitoring well drilling, sampling for a variety of media, reserve pit closures, demolition projects, and oil field investigations.

Education

Laramie High School, Laramie, Wyoming, 1963

Project Experience

- Site Supervisor/SSHO on Bristol Environmental Remediation Services, LLC, project for the U.S. Army Corps of Engineers (USACE), Alaska District for the N.E. Cape In-situ Chemical Oxidation (ISCO) and Intrusive Drum Removal/Landfill Cap, on St. Lawrence Island, Alaska. Directed the mobilization of a 30-man-camp and related heavy construction materials and equipment, via barge and landing craft, from Anchorage, Alaska to St. Lawrence Island, Alaska, which is located roughly 130 miles offshore west of the western coast of Alaska. The project conducted an In-situ Chemical Oxidation study on a subsurface hydrocarbon plume in arctic terrain and conditions. The project also included an intrusive removal of old drums containing waste oil that had been placed in a landfill, where the oil was recovered and the drums cleaned and reburied as inert debris in the landfill. The project included mining, hauling, and placing 28,000 cubic yards of cap material for the landfill and then revegetation of the landfill cap area. At the end of the project, all waste material, equipment, and camp were loaded on barges and demobilized. Job Value: \$6.2 million.
- Site Supervisor/SSHO on Bristol Environmental Remediation Services, LLC, project for the FAA to Clean and Inspect Diesel Fuel Tanks, Biorka Island and Level Island, Alaska. Supervised cleaning and inspection of diesel tanks and other activities. Responsibilities included SSHO duties. The scope of work included preparing planning documents and reports; mobilizing and demobilizing to and from Biorka Island; cleaning and inspecting five 20,000-gallon ASTs on Biorka Island; inspecting the secondary containment of the 20,000-gallon tanks; mobilizing and demobilizing to and from Level Island; and cleaning and inspecting two 10,000-gallon ASTs on Level Island. (2008)

- Site Supervisor/SSHO for the FAA Phase III Cape Yakataga Landfill Remedial Action. Supervised remedial action activities for Bristol Construction, LLC on FAA project. Responsibilities included SSHO duties. Phase III of this project is similar to Phase II, with the exception of additional confirmation sampling, site restoration, and demobilization. Phase II involved contaminated soil excavation, waste characterization, waste transportation, and disposal. Phase II was part of an increased scope of work, which extended Phase I to Phases II and III. Phase I consisted of debris and soil removal activities performed at the FAA Station at Cape Yakataga, Alaska. (April 2008)
- Site Supervisor/SSHO for the FAA Phase II Cape Yakataga Landfill Remedial Action. Supervised remedial action activities for Bristol Construction on FAA project. Responsibilities included SSHO duties. Phase II of this project was an extension of the scope of work of the Phase I debris and soil removal activities. Phase II involved contaminated soil excavation, waste characterization, waste transportation, and disposal. (2007)Site Supervisor/SSHO for the FAA Kodiak Air Traffic Control Tower (ATCT) AST Upgrades for Bristol Construction. Supervised AST replacement activities. Responsibilities included SSHO duties. The overall purpose of this project was to replace the existing ASTserving the ATCT at Kodiak, Alaska with a new 1,000-gallon capacity dual-wall AST currently under design by the FAA. (2007)
- Site Supervisor/SSHO for FAA Cold Bay AST Upgrades project. Supervised AST upgrade activities for Bristol Construction. Responsibilities included SSHO duties. (2007)
- Site Supervisor/SSHO for Biorka Island Groundwater Investigation. Supervised sampling and groundwater activities for Bristol Construction on FAA project. Responsibilities included SSHO duties. (2006)
- Site Supervisor/SSHO for the FAA Kodiak ATCT UST Upgrades. Supervised UST upgrade activities for Bristol Construction. Responsibilities included SSHO duties. (2006)
- Superintendent/SSHO, and Equipment Operator for Federal Aviation Administration (FAA) Adak Airport Tower Installation project. Directed a project that involved the upgrades of navigation aids at a Critical Navigation Site without the disruption of services. The scope of work included resealing two radomes by re-caulking and re-bolting (in excess of six thousand bolts and gaskets), demolition of two remote communication air/ground (RCAG) antennas and construction of two new RCAG antennas inside the radomes; the installation and burial of electrical and communications cables in over 300 lineal feet of trenches; the installation of two uninterruptible power supply systems (UPS); the construction of three new antennas (C-3, Glideslope, and Localizer); the repair of the main power supply box; and the installation of a new LCD lighting system on the NDB towers. The project also included installation of a new monitoring system, new piping, and the repair of an aboveground storage tank (AST) that furnishes fuel to the site emergency generator. The Project was at the remote Island site of Adak, Alaska. Job value: \$500,000. (September 2005)
- CQCSM for the U.S. Army Corps of Engineers (USACE), Alaska District, for the N.E. Cape Debris and Tram Demolition, St. Lawrence Island, Alaska. Set up the Project Quality Control and Site Safety Management System at the start of the fieldwork. Conducted all beginning of field project orientations and reparatory inspections. Conducted five safety classes for all-terrain vehicles per EM 385-1-1. Job value: \$5.2 million. (July 2005)
- CQCSM for CH2M Hill Constructors, Inc. (CCI), and Alternate SSHO for a U.S. Air Force project that involved capping an old landfill and constructing a new landfill with an adjoining asbestos cell. The project involved the excavation, placement, and grading of 112,000 cubic yards of three different soils types for the designed capping of the old landfill and

CHARLES L. (CHUCK) CROLEY

excavation of 80,000 cubic yards in the construction of the new landfill and asbestos cell. Job value: \$2.1 million. (May 2005)

- Superintendent, SSHO, and CQCSM for the Bureau of Land Management, and Equipment Operator for R & R Lodge Fuel Spill Cleanup. This project entailed excavation and sampling activities for a fuel spill from a fuel bladder and containment area at a remote hunting lodge in the Alaska Range. The project included the excavation of 55 cubic yards of fuelcontaminated soil over bedrock, alongside a short (1,600-foot) active airstrip, to a depth of 9 feet. Excavation was accomplished with small equipment. Five cubic yards of soil were removed from the site by small aircraft (Cessna 206) and 50 cubic yards were stockpiled on a liner for land-farming activities. Job value: \$100,000. (July 2004)
- Contract Site Supervisor for Chevron/Texaco for closure activities at an inactive reserve pit at West Kavik, a remote site on Alaska's North Slope. The first phase consisted of mobilization, construction, and demobilization of a remote site camp with Rolligons. The camp included power generation, freshwater treatment, grey water treatment, and cooking facilities, as well as living accommodations for 20 persons. The second phase consisted of mobilization and demobilization of equipment capable of mining approximately 8,500 cubic yards of gravel from an old airstrip and placing the gravel on top of an inactive reserve pit. Mr. Croley also acted as SSHO while he was on site. Job value: \$750,000. (February 2004)
- Superintendent/SSHO for a BLM project that consisted of demolition activities, a site investigation, and a historical site sampling activity for restoration at Red Devil Mine, a remote Alaska site where all equipment and personnel were mobilized by aircraft. The project included the demolition of six ASTs ranging from 200- to 350-barrel tanks and an ore hopper and ore-crushing facility. Project included the on-site burial of materials from demolition activities (including metal, wood, and concrete). Demolition activities took place in supplied air because of the presence of lead and mercury contaminants. A site investigation was conducted using a probe-pounding rig. A successful Historical Site Investigation was conducted for an ore house that had been destroyed more than 50 years prior and the site had been built over. The investigation was conducted using present-day air photos, old maps and field books, and a backhoe. Job value: \$450,000. (September 2003)
- Contract Field Operations Manager for Glenn Springs Holdings, Inc., a subsidiary of Occidental Petroleum, for a project that involved closure activities at three inactive reserve pits sites on the North Slope, Alaska. The first phase was the planning and mobilization of drilling equipment mounted on Rolligons to complete a subsurface investigation, and estimate drilling wastes and volumes of clean drill pad gravel. The second phase included the route selection and building and maintenance of eight miles of ice roads over tundra and river bottoms. The second phase also included the excavation and transport of 9,500 cubic yards of drilling wastes to the grind-and-inject facility at Prudhoe Bay from the reserve pit, and the hauling and placement of clean gravel, via Rolligon, at a third reserve pit. The work involved coordination among three oil companies and their contractors. Job value: \$1.25 million. (Winter 2002/2003).
- CQCSM/Alternate SSHO for the USACE, Alaska District. Managed demolition and site restoration of the Tok Fuel Terminal, Alaska. Site tasks included researching historical photographs; asbestos, polychlorinated biphenyls (PCBs), and lead-based paint (LBP) sampling; conducting a landfill investigation; construction of a solid waste landfill that included an asbestos cell; the removal and packaging of hazardous wastes; the removal of petroleum, oils, and lubricants (POL)-contaminated soil; site-wide abatement and disposal of asbestos and LBP; demolition and burial of 23 buildings; demolition and burial of four 1,000-gallon FSTs, one 1,000-barrel water storage tank, and one 5,000-barrel FST; and demolition

and removal of one 1,000-barrel FST, two 5,000-barrel FSTs, nine 30,000-barrel FSTs, and 30,000 lineal feet of tank-farm-related fuel and fire retardant pipelines. Job range: \$5 million+. (2001 to 2003)

- CQCSM/Alternate SSHO for the USACE, Alaska District. Managed the demolition of the Ben Eielson Taylor Elementary School, Eielson AFB, and the construction of an Olympicsized soccer field, a softball field, bleachers and fencing of the entire sports complex. Complicated demolition and disposal activities were involved, including security concerns with off-site disposal of debris, asbestos removal prior to demolition, and suspected mercury releases. Construction included leveling and placement of several types of soils, installation of an underground water hydrant system, concrete, asphalt, grass seeding, and fencing activities. Supervised quality control for contractor and subcontractor activities. Job range: \$1.2 million. (2001)
- CQCSM/Alternate SSHO for the USACE, Alaska District. Managed multifaceted demolition of a long-range radar station at a U.S. Air Force site in Fort Yukon, Alaska. Directed removal and long-term storage of more than 650 cubic yards of POL-contaminated soils. Supervised asbestos removal and asbestos storage of materials from 13 buildings, four radar towers, and utility facilities; demolition of two 60-foot by 60-foot and two 120-foot by 120-foot radar towers; demolition and debris removal of 12 buildings; decommissioning and demolition of 26 ASTs; construction of a solid waste landfill; placement of various types of demolition debris in the landfill, including use of an asbestos cell; and capping of the landfill to State of Alaska criteria. Conducted soils exploration program and water sampling; constructed new fuel storage and monitoring system. Installed biovent system. Job value: \$5 million. (1999 to 2001)
- CQCSM/SSHO for USACE, Alaska District/FAA. Managed FST upgrades at Port Heiden, Wrangell, Metlakatla, Sand Point, and Dillingham, Alaska (1998). Project entailed removal of seven regulated underground storage tanks (USTs) and one AST, and installation of five ASTs for prime fuel sources at remote navigation aid sites. Fuel systems included lead detection, inventory control, and remote site monitoring systems. Responsibilities included on-site construction management and health and safety, developing reporting documents, and assisting in planning and submittal of documents.
- CQCSM/SSHO for the Galena (Alaska) Air Force Station (AFS) Tank Removal and Soil Remediation Project for the USACE (1997). Managed cleaning of three bulk fuel ASTs; decommissioning of three USTs; and construction, operation, and maintenance of a 5,100-cubic-yard bioremediation cell. The project included demolition, asbestos abatement and waste management. Responsibilities included on-site construction management and assisting with completing planning and reporting documents, managing submittals, performing network analysis, and submitting pay requests.
- CQCSM/SSHO for the USACE at the Galena AFS, Alaska, Power Plant (1996 to 1997). Managed removal of two 12,000-gallon and two 25,000-gallon fuel USTs and five 55- to 1,000-gallon USTs that contained fuel and oil/water separator waste; removal and stockpiling of 700 cubic yards of contaminated soil; installation of two 30,000-gallon ASTs at a remote site off the road system. Responsibilities included on-site construction management, site safety, and assisting with completing planning and reporting documents, managing submittals, performing network analysis, and submitting pay requests.
- Site Superintendent for Exxon Mobil, Alaska. Provided construction and safety oversight and permit compliance for closeout of two inactive reserve pits at Flaxman Island on Alaska's North Slope. Winter 2001 activities included drilling a new 2,500-foot disposal well for grinding and injecting reserve pit wastes; excavation of two inactive reserve pits and two

flare pits; confirmation sampling and on-site laboratory analyses; slurrying and injecting cuttings; and reviewing and verifying quantities and pay items. Winter 2002 activities included construction of a 68-mile offshore ice road on the Arctic Ocean; excavation of contaminated soil from reserve pits, and the excavation and hauling of 20,000 cubic yards of drilling wastes to the Prudhoe Bay grind and injection facility. Project considerations included sensitive wildlife habitats, construction in arctic conditions, and North Slope safety requirements. Job range: \$7.5 million.

- Contract Site Quality Control Manager for the Alaska Department of Natural Resources, Joint Pipeline Office (JPO) for the Northstar Development Project, Point McIntyre/Point Storkerson, North Slope, Alaska. Provided in-field quality assurance monitoring for BP Exploration (Alaska), Inc., during construction of two 10-inch pipelines running from Seal Island, offshore, to Point McIntyre, onshore, and then onshore and terminating at BP's Gathering Center 1. The offshore underwater pipeline portion was approximately 6 miles long and depths to 50 feet.
- Site Superintendent for Exxon Company, USA, for a cleanup project at a former fuel storage area at the Alaska State A-1 drill site on remote Flaxman Island, in the Beaufort Sea. The project involved the use of a field laboratory to field screen and segregate 1,000 cubic yards of soil during the winter. The excavated contaminated soil was then transported, via Roligon, back to the Prudhoe Bay area for treatment.
- Site Superintendent for Exxon Company, USA, on a project that consisted of winter investigations of two inactive reserve pits at Alaska State A-1 and G-2 drill sites on Flaxman Island, Alaska, a remote Island in the Beaufort Sea. The investigations included relocation of the reserve pits, soil drilling with a drill rig transported via Roligon, excavation of trenches (in permafrost materials) for drill mud sampling and investigating the use of liners.
- Contract Site Quality Control Manager for the Alaska Department of Natural Resources, JPO for the Alpine Development Project, Colville River, North Slope, Alaska. Provided infield quality assurance monitoring during horizontal directional drilling and installation of four pipelines beneath the Colville River. The crossing was approximately 4,100 feet long.
- Construction Manager/SSHO for USACE project at Fort Wainwright, Alaska. Provided construction management of an experimental soil gas recovery system that included the installation of two horizontally drilled wells, a 1,000-foot-long air-injection well, and a 750-foot-long vapor-extraction well. The experimental system included the installation of a variety of monitoring wells and nuclear density probe wells, as well as the compressor plant for the air injection. Also implemented site safety plan.
- Construction Superintendent/SSHO for FST improvements for the FAA in McGrath, Alaska. Supervised project to decommission eight FSTs and install seven FSTs. Also responsible for site safety.
- Construction Superintendent/SSHO for FST improvements for the FAA in Bethel, Alaska. Supervised the decommissioning of 14 FSTs and installation of 9 FSTs. Also responsible for site safety.
- Construction Superintendent/SSHO for the FAA for a project in Cordova, Alaska. Supervised the decommissioning of 19 FSTs and installation of nine FSTs. Responsible for site safety.
- Construction Superintendent/SSHO for the Municipality of Anchorage. Directed field operations for decommissioning of three USTs at a power-generating facility.

- Construction Superintendent/SSHO for the FAA. Directed field operations for the FAA for Alaska (statewide) FST replacement project to decommission USTs and ASTs, construct new fuel systems, and clean up fuel-affected soil. Responsible for site safety. Completed projects at four Anchorage and 16 rural locations, involving 190 USTs and ASTs, 122 decommissionings, 79 installations, and 11 upgrades (1990-1998).
- Senior Technician for the Hunters Point Annex Restoration in San Francisco, California. Logged borings, field-screened soil samples for radiation, installed and sampled monitoring wells, located drill borings for future projects, and mapped dump sites suspected of containing radiation-affected waste.
- Drilling Superintendent/Senior Technician for the FAA at Bettles, Alaska. Performed groundwater investigations. Supervised drilling and environmental soil and water sampling program to trace the limits of a contaminant plume. Responsible for site safety.
- Drilling Superintendent/Senior Technician for a confidential client in Kenai, Alaska. Supervised a reserve pit monitoring project over a two-year period. Supervised field operations including drilling, environmental soil sampling, and groundwater testing for possible groundwater contamination.
- Drilling Superintendent for the Milne Point Gravel Study, North Slope, Alaska, for Conoco, Inc. Directed a drilling and soil sampling program for gravel mine site exploration.
- Drilling Superintendent for Point McIntyre Development, North Slope, Alaska, for ARCO Alaska, Inc. Supervised a drilling and soil sampling program for a foundation study for a drill pad design and pipeline construction. Installed a ground temperature monitoring system. Drilling activities included onshore and over-ice operations.
- Drilling Superintendent for Sohio Petroleum Company. Supervised field investigation for the Endicott Geotechnical Investigation, Beaufort Sea, Alaska, which involved drilling onshore and offshore soil borings, and performing in-situ testing to establish design criteria for the development of Endicott oil field facilities. Coordinated field crews, maintained all equipment, and troubleshot drilling problems.
- Superintendent/Senior Technician for ARCO Alaska, Inc., U5-A Slab Investigation, North Slope, Alaska. Supervised drilling for an environmental soil sampling and geotechnical drilling program inside a warehouse in a permafrost area. The purpose of the project was to investigate a foundation failure and related chemical release.
- Superintendent for the USACE Hazardous, Toxic, and Radioactive Waste Program at various sites throughout Alaska. Served as drilling superintendent for FST decommissionings and installations, soil and water investigations and studies, and remedial action and construction projects.
- Senior Technician for the USACE, Sacramento District, at Fort Ord, California. Performed remedial investigation for the installation and sampling of monitoring wells, and collection of inventory and control samples.
- Senior Technician for ARCO Alaska, Inc. Developed a system to sample for heavy metals in high-pressure natural gas at Prudhoe Bay, Alaska.
- Senior Technician for Exxon Company, U.S.A. Conducted environmental soil sampling programs on and around contaminated soil stockpiles in Valdez and Seward, Alaska.
- Senior Technician for confidential client. Conducted environmental soil sampling programs on a soil bioremediation project near Beluga, Alaska. The sampling took place at several

remote gravel pads in southcentral Alaska. Directed the initial construction of two bioremediation cells.

- Senior Technician for Exxon Company U.S.A., Tatitlek Soil Remediation Project, Southcentral Alaska. Directed environmental soil sampling programs at two remote sites to support and document site cleanup.
- Senior Technician for Chevron U.S.A., Inc. Directed drilling operations for sampling the core of a man-made ice island and constructing a monitoring system in the Beaufort Sea, Alaska. Conducted over-ice sampling for future ice or gravel island drilling locations.
- Senior Technician for remote Alaska site for the State of Alaska, in Minto, Alaska. Responsible for overseeing groundwater investigation and permanent abandonment of a freshwater production well.
- Senior Technician for ARCO Alaska, Inc. Directed drilling operations and recovery of seismic equipment, and construction of a seismic monitoring system for a production well test (UGNU tiltmeters) on the North Slope, Alaska.
- Senior Technician for ARCO Alaska, Inc., and Conoco, Inc. Directed drilling and environmental soil sampling for reserve pit closeout permit requirements on the North Slope of Alaska, using hollow-stem auger and coring systems. Installed permanent ground temperature monitoring systems. Collected and field tested surface-water samples to monitor closeout permit compliance.
- Senior Technician for Exxon Company, U.S.A. Conducted drilling and sampling programs at a remote arctic exploration site (Point Thomson Units 1 and 4, North Slope, Alaska) during summer and winter. Directed bioremediation activities at the same site, including mobilization and demobilization of workers, equipment, camp facilities, and bioremediation work, using marine and overland transportation.
- Senior Technician for project for the Municipality of Anchorage's Alaska Aviation Heritage Museum. Responsible for overseeing the removal of three USTs in a shallow groundwater area.
- Senior Technician for a confidential client in Anchorage, Alaska. Performed service station site investigation and directed drilling operations for soil testing around buried facilities and utilities.
- Senior Technician for the Municipality of Anchorage (Alaska). Drilled five offshore borings and performed cone penetrometer tests for a causeway linking Anchorage and Fire Island.
- Senior Technician for the Third Avenue Shelter project for the Municipality of Anchorage (Alaska). Drilled three borings in an earthquake slide area in which cone penetrometer testing was conducted to a depth of 120 feet.
- Senior Technician for the Municipality of Anchorage (Alaska). Participated in the following area projects:
 - Peters Creek Watershed Improvement District (W.I.D.) 337
 - Nancy Local Improvement District 174 and W.I.D. 353
 - Chester Creek Oil and Gas Separators
 - West 42nd Avenue
 - West High Culvert

- 56th Street Walls
- Girdwood Anchorage Telephone Utility Site
- 39th and 40th Streets, Anchorage Telephone Utility Site
- Southeast Interceptor Project
- Bear Valley Anchorage Telephone Utility Site
- Chugiak Fire Station
- Hiland Drive Slope Stabilization
- Dimond Trunk Storm Drainage Study
- Senior Field Technician/Drilling Superintendent for ARCO Alaska, Inc. Performed geotechnical investigation for Prudhoe Bay Unit reserve pits on the North Slope of Alaska. Work consisted of drilling and logging test borings via 3-inch frozen cores. Project objective was to measure the depth of chemical contamination beneath the reserve pit. Collected soil samples for chemical analyses.
- Senior Field Technician/Drilling Superintendent for Union Oil Company of California project. Performed groundwater investigation on the Kenai Peninsula, Alaska. Assisted in drilling borings and sampling soil and groundwater for geochemical analyses to evaluate impacts on groundwater resources and potential contaminant transfer.
- Senior Field Technician/Drilling Superintendent for Butler Aviation project, Anchorage, Alaska. Performed site background investigation. Drilled borings and sampled soil and groundwater for geochemical laboratory analyses.
- Senior Field Technician/Drilling Superintendent for ARCO Alaska, Inc. Performed work on an environmental project on the North Slope of Alaska, to explore possible effects of dispersion and biological accumulation of chemical contaminants in tundra. Duties included sampling surface water, soil, and vegetation at 250 sampling points for geochemical analyses. Assisted in field measurements of pH, electrical conductivity, and dissolved oxygen content of water.
- Senior Field Technician/Drilling Superintendent or USKH, Inc. Performed site investigation at the U.S. Coast Guard Aleutian Air Station Detachment. Participated in drilling borings, sampling soil and water, conducting geophysical investigations, and monitoring groundwater.
- Senior Field Technician/Drilling Superintendent for ARCO Alaska, Inc. Performed an investigation to examine the potential for reserve pit water to seep through gravel containment berms on the North Slope, Alaska. Assisted in installing and monitoring instrumentation to identify groundwater characteristics in saturated and unsaturated zones, and to profile ground temperatures. Collected groundwater, soil, reserve pit water, and drilling reserve samples for geochemical analyses.
- Senior Field Technician/Drilling Superintendent for a confidential client. Performed multiphase investigation of impacts of plant discharges on groundwater in a multi-aquifer system for the Bernice Lake Power Plant in Alaska. During the initial phase, performed geochemical sampling of groundwater to evaluate potential problems. In Phase II, assisted in installing and monitoring groundwater and ground temperature instrumentation.

CHARLES L. (CHUCK) CROLEY

- Senior Field Technician/Drilling Superintendent for Tesoro Alaska Petroleum. Performed soil and groundwater contamination investigation for an underground hydrocarbon spill at an industrial facility. Participated in drilling test borings and sampling soil and groundwater.
- Senior Field Technician/Drilling Superintendent for Pacific Gas and Electric's Hinkley Compressor Station in Hinkley, California. Performed preliminary site appraisal and participated in collecting groundwater samples from approximately 100 wells including domestic, agricultural, public water supply, and industrial wells in an investigation of chromium-contaminated groundwater.
- Senior Field Technician/Drilling Superintendent for ARCO Alaska, Inc. Installed thermistors in closed-out reserve pits and in an active waste oil reserve pit on the North Slope of Alaska.
- Senior Field Technician/Drilling Superintendent for ARCO Alaska, Inc. Performed geotechnical investigation project, sampled soil, performed resistivity testing, and installed thermistors as part of freeze-thaw studies to redesign a flare pit on the North Slope, Alaska.
- Senior Field Technician/Drilling Superintendent for America North, Inc./Alaska Gold. Drilled borings for the Steadman Field Site Investigation, and sampled soil contaminated with mercury and arsenic in Nome, Alaska. Project included investigating a waste disposal area.

Other related project experience includes the following:

- Duck Island Development Area, Beaufort Sea, Alaska
- Port of Nome Over-Ice Investigation, Nome, Alaska
- Soil Boring Programs, Trans-Alaska Pipeline Route
- Mukluk Island Site, Beaufort Sea, Alaska
- Offshore Drilling, Beaufort Sea, Alaska
- Drilling of Five Island Sites, Beaufort Sea, Alaska
- Wharf and Docking Facilities, Afognak Island, Alaska
- Rotary Drilling and Wireline Coring, Remote Island in Indian Ocean
- Alpine Permafrost Institute, Pikes Peak, Colorado

Professional Experience

- CQCSM/Construction Superintendent, Bristol Environmental & Engineering Services Corporation. Responsible for CQCSM to close existing municipal solid waste landfill and construction of a new landfill at Eareckson Air Station on Shemya Island, Alaska, on a subcontract to CH2M Hill. Also responsible for CQCSM role for a removal action to demolish and dispose of a tram line and waterlines at the former White Alice Communications Site at Northeast Cape on St. Lawrence Island, Alaska, for the USACE. Involved in ongoing FST project work for the FAA. (2004 to present)
- Driller, Senior Technician, Drill Superintendent, Construction Superintendent, and Field Operations Manager for MACTEC Engineering and Consulting Inc., and its predecessors (Harding ESE and Harding Lawson Associates). Also, performed the role of CQCSM and alternate SSHO on many USACE Projects throughout Alaska. (1979 to October 2004) Description of duties in the various positions are as follows:
 - As senior technician, responsibilities included installing monitoring wells; sampling water and soil; handling oil and hazardous substances; performing field measurements on

water samples; installing soil-gas wells; and installing thermistors, manometers, and piezometers. Conducted freeze-thaw studies, cone penetrometer tests, permafrost investigations, and percolation tests.

- As general drilling superintendent, operated and maintained drilling equipment, supervised drill crews, and was responsible for site safety. Experienced with permafrost drilling, refrigerated coring, mineral exploration, dam foundation drilling and testing, overwater and over-ice operations, and helicopter drilling.
- As construction superintendent, mobilized and demobilized construction crews and materials to various remote Alaska sites via air, land, and water transportation. Provided oversight for removal and storage of contaminated soil, decommissioning of USTs and ASTs, and installation of new FSTs and distribution systems, and was responsible for site safety.

Certifications and Training

Certified UST Worker, State of Alaska (Installation/Retrofitting and Decommissioning) Alaska No. 172 Certified in UST Installation/Retrofitting, International Code Council No. 1057168-U1 Certified in UST Decommissioning, International Code Council-No. 1057168-U2 Certified in the Use of Nuclear Testing Equipment - Alaska No. 16619 Certified Safety Instructor-ATV Safety Institute-ID No. 120099 U.S. Environmental Protection Agency (EPA)/Asbestos Hazard Emergency Response Act AHERA-Asbestos Abatement Worker - Alaska No. 5249 (Current since 1989) 30-Hour OSHA Construction Safety and Health 40-hour EPA/AHERA Asbestos Supervisor/Worker, plus 8-hour Refresher 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER), plus 8-hour Supervisor and 8-hour Refresher, Bristol Industries CPR and First Aid for Adults, MEDIC FIRST AID[®] International 8-hour Entry to Confined Spaces 24-hour Excavation, Trenching, and Soil Mechanics 24-hour Construction Project Administration Hydrogen Sulfide Safety Training Radiation Protection Training USACE Construction Quality Management for Contractors 24-hour Hazardous Materials Transportation 10-hour Construction Safety **Defensive Driving Training**

RUSSELL C. JAMES

Environmental Scientist

Areas of Expertise

- Environmental Sampling and Monitoring
- ✓ Technical Writing
- ✓ GIS
- ✓ GPS
- ✓ Database Management
- ✓ ANCSA Land Mapping

Mr. James has 6 years of experience in demonstrating proficiency and expertise in Geographic Information Systems (GIS) and Geographic Positioning Systems (GPS). He has integrated GPS and GIS for a number of projects with government agencies and private organizations, and is adept at combining GIS/GPS with environmental sampling and geologic mapping. He is well versed in databases and skilled in the use of ArcGIS, Geomedia Professional, and Trimble® GPS equipment and software. Mr. James has performed environmental field work in Alaska, Arizona, and New Mexico. He is experienced in collecting soil, sediment, and water samples; soil boring and monitoring well installation; underground storage tank removal; conducting Phase I Site Assessments; and technical writing.

Education

B.S., Environmental Geography; Minor, Geology, Valdosta State University, Valdosta, Georgia, 2005

Project Experience

- Construction Quality Control Systems Manager (CQCSM) and Environmental Scientist for Northeast Cape In-Situ Chemical Oxidation (ISCO) Study and Intrusive Drum Removal/Landfill Cap (2009). Responsibilities include ensuring contract specifications between the U.S. Army Corps of Engineers (USACE) and Bristol; providing oversight for various activities performed in the field; and acting as liaison between Bristol and USACE. Tasks involved daily reporting to USACE, GPS, and GIS mapping services, meeting with subcontractors, reporting to the Bristol home office, environmental sampling, authoring planning documents, and writing the Removal Action Report.
- GIS Specialist and field data collection personnel for monitoring well inventories on Fort Wainwright and Fort Richardson (2009). Responsibilities include updating the current database regarding monitoring wells, maintaining open communications with the USACE's GIS point of contact, and establishing effective field data collection techniques using GPS. The project goal is to implement a more effective and accurate GIS database regarding the status and position of monitoring wells on base. Tasks included GPS field collection, and data management and integration into USACE's GIS standards.
- GIS Specialist for Alaska Natural Gas Development Authority Wetlands Delineation & Project Management project (2008). Project responsibilities included prepping data and GPS units for field crews; maintaining and organizing GPS field data; and displaying field data in GIS and map atlases, which consisted of hundreds of alignment sheets encompassing over 350 miles of potential pipeline corridor.
- Environmental Scientist for three potential Leaking Underground Storage Tank (LUST) sites in the Navajo Nation and EPA Region 9 (2008). Assisted in the supervision of subcontractors excavating Underground Storage Tanks (USTs) for removal. Six USTs were

removed from three sites. Collected field screening headspace samples using a photoionization detector (PID). Collected soil and surface water samples for analysis at fixed laboratory.

- Environmental Scientist for FAA Cape Yakataga Landfill Removal project, Phase III, Cape Yakataga, Alaska (2008). Collected waste characterization and confirmation soil samples for the decommissioning of a landfill and biocell. Monitored the installation of soil borings and monitoring wells, and conducted groundwater sampling. Authored final report summarizing field activities, presenting analytical data, and providing recommendations for future site remediation.
- Fort Richardson UST Corrective Action, Anchorage, Alaska (2007). Project responsibilities include split spoon sample collection, soil boring oversight, soil classification, and acquisition of dig permits. Collected field-screening headspace samples using a PID.
- Environmental Scientist for FAA Unalakleet Release Investigation, Unalakleet, Alaska (2007). Acquired surface and subsurface soil samples from eight sites near Unalakleet, Alaska. Collected field-screening headspace samples using a PID. Also conducted fieldscreening using Horiba OCMA 350 Infrared Spectrometer.
- Environmental Scientist providing project support for Elmendorf Treatability Study, Anchorage, Alaska (2007). Assisted installation of bladder pump and set up of micro purge system for groundwater sampling from monitoring wells. Calibrated YSI brand water quality meter and logging system for groundwater monitoring. Helped with construction of well injection system.
- Environmental Scientist for FAA Cape Yakataga Landfill Removal project, Phase II, Cape Yakataga, Alaska (2007). Responsible for soil sample collection; waste container data management, and packaging and shipping of soil samples. Composed interim progress report and authored work plan for 2008 field activities.
- Environmental Scientist for Hanna Dimond Project, Anchorage, Alaska. Project responsibilities include collecting water samples from aboveground tanks, and soil samples from stockpiles. Collected field-screening headspace samples using a PID.
- Environmental Scientist for 4th & Gambell Streets Project, Anchorage, Alaska. Project responsibilities included installation of soil borings, soil classification, split-spoon sample collection, oversight of monitoring well installation. Collected field-screening headspace samples using a PID.
- Environmental Scientist for Phase I Environmental Site Assessments at three sites in Anchorage, Alaska. Project responsibilities included conducting site visits and interviews, database searches, and preparation of report and figures.
- GIS Specialist for CAMPTEX Project, Bristol Bay Region, Alaska, for Bristol Bay Native Corporation (BBNC). Project responsibilities include organizing, analyzing, and maintaining GIS data; acquiring knowledge about the Alaska Native Claims Settlement Act (ANCSA), and adding/digitizing BBNC ANCSA lands into GIS using Geomedia.
- Tift County Board of Education Campus Mapping Project, Tift County, Georgia. Responsible for GPS collection of utility points, post-processing analysis of GPS in ArcGIS, and digital production of gas, water, and sewer lines. Involved in acquisition and georeferencing of 14 school floor plans. Nominated for 2006 National Association of Development Organizations (NADO) Innovation Award.

Russell C. James

- ◆ City of Douglas Utility Mapping Project, Douglas, Georgia. Responsible for the GPS collection and post-processing of utility points contained within public rights-of-way. Points collected include street lights, manhole covers, stormwater collection inlets, fire hydrants, water meters, water valves, gas valves, and gas meters, etc. Points were collected with a Trimble GeoXT[™] mounted onto a bicycle, post-processed in Pathfinder[®] Office, and combined into a GIS using ArcMap.
- Cook County Emergency 911 Address Mapping Project, Cook County, Georgia. Responsible for the GPS collection of every address "point-of-entry" within the limits of Cook County. Points were collected with a Trimble ProXR GPS and combined into a GIS using ArcMap 9.1.
- City of Tifton Utility and Right-of-Way Mapping Project, Tifton, Georgia. Responsible for GPS collection of utility points within public rights-of-way in the city of Tifton, Georgia. Points were collected using Trimble ProXR backpack unit and bicycle mount.
- Thomas County Sign and Bridge Inventory, Thomas County, Georgia. Responsible for the GPS collection of signs and bridges along every county maintained road in Thomas County.

Professional Experience

- Environmental Scientist for Bristol Environmental Remediation Services, LLC (2007 to present). Responsible for GIS mapping and data collection; conducting site assessments and site investigations; writing technical reports; and performing environmental sampling and monitoring, including soil borings, well installations, research, and data collection.
- GIS Specialist for Bristol Environmental & Engineering Services Corporation (BEESC) (November 2006 to July 2007). Responsibilities included data compilation, organization, and production of BBNC and BEESC GIS data.
- GIS Data Collector for South Georgia Regional Development Center (December 2003 to September 2006). Responsibilities included GPS collection of field data, analysis and presentation of data in GIS, as well as maintenance and training for Trimble GPS units and software.
- Geology Research Intern, 2004 ACRES Program, Georgia State University. Analyzed the geochemistry of metamorphic rocks in the Uchee Belt, near Columbus, Georgia. Utilized ICP-MS and XRF for chemical analyses of prepared samples. Poster presentation at the Annual GSA Meeting in Denver, Colorado. Abstract can be found at <u>http://gsa.confex.com/gsa/2004AM/finalprogram/abstract_79798.htm</u>.

Training and Certifications

Certified Erosion and Sediment Control Lead (CESCL) - March 27, 2009

U.S. Army Corps of Engineers, Construction Quality Management For Contractors – April 11, 2008

HAZWOPER Supervisor Training – March, 2008

8-Hour HAZWOPER Training – March 4, 2009

CPR and First Aid for Adults – February 19, 2008

40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) - July 20, 2007

Hazardous Materials Transportation (DOT/IATA) – February 14, 2008

Basic CPR and First Aid for Adults - February 19, 2008

Software Capabilities

MS Office 2007, including Word, Excel, PowerPoint, and Access, gINT, GeoMedia Professional v6.1, ArcGIS v9.3, ArcPad 8, ER Mapper, GPS Pathfinder Office, Trimble TerraSync, Visual Sample Plan v5.0.

Awards

Outstanding Service Award, South Georgia RDC, 2006 Honor Graduate: Magna Cum Laude, 2005 Outstanding Student in Environmental Geography, 2005 Gertrude Odum Scholarship, 2000-2004 HOPE Scholarship, 2000-2004

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MARTIN (MARTY) HANNAH

Environmental Scientist/Project Chemist

Areas of Expertise

- ✓ Environmental Chemistry
- ✓ Toxicology
- Environmental Site Investigations
- ✓ Quality Assurance/Quality Control
- ✓ Site Remediation

Mr. Hannah has over 12 years of environmental experience, including 4 years in environmental remediation. His expertise encompasses site assessment and remediation projects, site investigations, guality assurance (QA)/quality control (QC) requirements, and project chemistry. He has worked on projects for federal and state agencies and is familiar with the standards and procedures for compliance with these agencies. Mr. Hannah's expertise includes management and transportation of hazardous waste materials at remote arctic project sites. In addition, he has served as Research Professional/Laboratory Manager for the University of Alaska Anchorage School of Engineering, and has been responsible for all aspects of a scientific field equipment business as the sole proprietor of Hannah Instrumentation

Education

B.S. - Biology, Emphasis in Toxicology, Chemistry and Emergency Medicine,

Mankato State University, Mankato, Minnesota 1992

M.S. - Environmental Quality Science, *Emphasis on Remedial Feasibility Studies*, University of Alaska Anchorage, 2005

Project Experience

- Field Chemist, Nome, Alaska (2009). Performed environmental assessment of mercury and arsenic contamination at a former gold processing facility. Duties included creation of a work plan, sample and analysis plan, and procedures for field analysis of mercury (mobile laboratory). Performed analysis of soil samples on site to delineate the extent and concentration of mercury contamination. Directed drillers on continued sample collection based on field analytical results.
- Environmental Scientist, Spill Response near the Iliamna River, Alaska (2009). Provided support to client in response to a fuel spill near the Iliamna River. Coordinated client personnel in spill response and remediation of contaminated soil. Collected soil and surface water samples. Advised client on regulatory requirements and submittals to State agencies, as well as development of remedial methods for reduction of contaminants in impacted soils.

Project Chemist, Northeast Cape, St. Lawrence Island, Alaska (2009). Provided support to field activities at Northeast Cape for remedial pilot tests and removal of contaminants at a Formerly Used Defense Site (FUDS). Coordinated submittal of samples and evaluated laboratory data for quality and representativeness to the site. Functioned as the primary point of contact for fixed lab, project managers, and field personnel regarding procedures and submittal of samples for analyses. Responsible for laboratory reports and electronic data deliverables.

- Environmental Scientist/Remediation Specialist, Elmendorf Air Force Base (AFB) (September 2006 May 2009). Supported monitoring, and operation and maintenance of several remedial systems, including sites located within the active airfield and numerous other sites on the installation. Responsible for dig permits, well installation and decommissioning, soil borings, and soil gas vapor analysis, along with operation and maintenance of bioventing systems.
- Field Chemist/Environmental Scientist, USACE, POL-Contaminated Soil Remediation, Umiat, Alaska (July – August 2006). Collected field and confirmation soil samples using multi-incremental sampling (MIS) on thermal infrared (IR)-treated soil. Prepared the methods, testing, instrumentation, and environmental controls for field analysis of samples by U.S. Environmental Protection Agency (EPA) Method 1664. Coordinated the shipping of rush samples, equipment, and materials to and from this remote arctic site.
- Environmental Scientist, QA/QC Officer, Environmental Data Manager BP Exploration (Alaska) on several Site Assessment and Remediation Contracts (2006 2009). North Slope, Alaska. Provided QA and procedural input in the development and release of an extensive overhaul of BP's environmental Quality Assurance Program Plan.Project Chemist for Oasis Environmental Responsible for laboratory data management, QA program plans, final review and validation of laboratory data on numerous Alaska Department of Environmental Conservation (ADEC), Federal, and private projects. Additional responsibilities included completion of ADEC laboratory data checklists, quality of analytical reviews, and flagging of tabulated data.
 - Field Scientist Performed monitoring and remedial action on former and existing Chevron gasoline stations and bulk fuel plants in Anchorage and Fairbanks, Alaska. Performed as Field Lead on soil, groundwater, and surface water sampling events. Supported implementation and operation of remedial systems. Responsible for operation and maintenance of existing remedial systems. Treatment technologies included soil vapor extraction, air sparging, granular activated carbon water treatment, and freeproduct recovery using high-vacuum extraction.

Professional Experience

- Environmental Scientist/Project Chemist, Bristol Environmental Remediation Services, LLC. Responsible for Initial project proposal and attention to cost control preparation of sitespecific DQOs with SAP and QAPP documentation, contract negotiation, master service agreements, invoice tracking and coordination of field team, providing oversight of collection to laboratory delivery data reduction, and presentation of the site contamination and riskbased calculations, data validation QA/QC effort, including ADEC and AFCEE ERPIMS submittals.
- Project Chemist, Environmental Scientist, HM & DG Shipping Specialist, Field Equipment Manager, OASIS Environmental, Anchorage, Alaska (2006 – 2009). Responsible for Quality Assurance Program Plans, standard field procedures, and management of laboratory data. Managed, shipped, and serviced all scientific monitoring instrumentation and support equipment for OASIS' five offices. Equipment included photoionization detectors (PIDs)/flame-ionization detectors, multi-gas meters, water quality multi-meters, pumps, and a wide variety of other field equipment.
- Hannah Instrumentation Owner-Sole Proprietor, Anchorage, Alaska (1998 2009). Responsible for all aspects of a scientific field equipment business that leases PIDs, multigas meters, water quality multi-meters, pumps and other equipment used by personnel performing site investigations, and monitoring and remediation services. Provided analytical

Martin (Marty) Hannah

equipment and chemical analysis support for mobile laboratory operations using gas chromatographs, IR spectrophotometers, and other field instrumentation for quantifying a wide variety of contaminants of concern.

- Field Services Coordinator, North Creek Analytical (1999 2004), Anchorage, Alaska. Duties included support for clients and laboratories for all aspects of environmental sampling and analyses for contaminants of concern. Performed tasks such as filling client bottle orders, receiving samples, and forwarding them to the proper laboratories within specified temperature and packing regulations. He also provided support to NCA mobile laboratories in Amchitka, Adak, Prudhoe Bay, and Livengood, Alaska.
- Organic Chemist/GC Analyst Semivolatiles for Columbia Analytical Services, Anchorage, Alaska (1993 – 1998). Performed analyses of environmental samples on various matrices for contaminants of concern such as fuels, poly-chlorinated biphenyls (PCBs), pesticides and PAHs. Performed maintenance and repair of gas chromatographs and data systems. Managed waste stream and led effort to reduce the hazardous waste generation. Other duties included supporting laboratory personnel in compliance with Federal, state and municipal regulations for safety and other code compliance.
- Organic Chemist/GC Analyst, Analytica Alaska (1992 1993). Performed analyses on soils and waters for Alaska and EPA methods AK101 and EPA 8021B (GRO/BTEX).
- Research Professional-Laboratory Manager, University of Alaska Anchorage, School of Engineering (1998 2005). Responsible for all aspects of physical and research laboratories, including all health, safety, and environmental (HSE) policies and procedures in teaching and research laboratories. Maintained chemical inventories and instructed researchers and graduate students in proper handling of chemicals and operation of various physical and analytical systems and instrumentation. Performed numerous tasks either solely or in support of environmental remediation feasibility studies on contaminated soils and waters.
- Assistant Laboratory Manager of the Applied Science and Engineering Technology (ASET) laboratory, which utilized state-of-the-art instrumentation in support of chemistry, biology, and engineering research. Duties included selection, procurement, installation and operation of the instrumentation, as well as ancillary personal protective equipment. Prepared Standard Operating Procedures for the operation of analytical instrumentation and analysis using a wide variety of analytical methods used in the laboratory.

Professional Publications

Magnitude and Variability of Biogenic Interference in Cold Regions Soils. Journal of Cold Regions Engineering, September 1999. C.R. Woolard, D.M. White, J.L. Walworth, M.E. Hannah.

Software Capabilities

Microsoft Office Products

ProUCL (EPA risk assessment)

Various data logger programs and software, such as UCON and PC2001

Training and Certifications

EPA 40-hour HAZWOPER

EPA 8-hour HAZWOPER refresher, current

Emergency First Aid and CPR

DOT/IATA Dangerous Goods Shipper's Training

USAF Flight Line Training-Elmendorf AFB

BP North Slope Red Book Training for handling waste generated on the North Slope

Smith Safe Driving Course-Provided by BP Exploration Alaska

ERIC BARNHILL

Environmental Scientist

Areas of Expertise

- ✓ Biology
- ✓ Fisheries Research
- ✓ Research Development
- ✓ Remedial Investigation Sampling
- ✓ Groundwater Sampling

Mr. Barnhill is a well-rounded biologist with 12 years of research and administrative experience in environmental science and contaminated sites projects, including site assessments and groundwater monitoring investigations. Mr. Barnhill has an extensive background in fisheries science, including both the research and the development sides of numerous fisheries projects. Additionally, he has been responsible for developing contracts and research plans for fisheries research. His end goal has been support of continued sustainability of Alaska's fisheries resource and the areas in which they inhabit. Among his many attributes, he has proficient skills in public speaking.

Education

B.S., Biology, Eastern Washington University, 1999

Project Experience

- Environmental Scientist for Bristol Environmental Remediation, LLC (March 31, 2008 present).
 - Environmental Scientist for Selawik Soil Sampling project. The project consisted of collecting confirmation samples of soil from underneath an aboveground storage tank (AST) where an overfill of two gallons of diesel fuel occurred years earlier. Duties included sample taking in frozen soil, packing and shipping of samples, and swing tying.
 - Environmental Scientist acting as field data collection personnel for Fort Wainwright Operating Unit 3 (2009). Responsibilities included collecting well information and taking groundwater parameters for DRO, GRO, VOC, EDB, PAH, iron (II), lead, and sulfate analysis using low-flow groundwater sampling techniques.
 - Environmental Scientist for Fort Richardson Well Inventory project (2009). Responsibilities included researching information on well locations, physically finding wells using Trimble GPS unit, and taking well field parameters, including well casing size, depth of well, depth to water and taking GPS positions for inclusion in a GIS database.
 - Lead Environmental Sampler for Northeast Cape In-Situ Chemical Oxidation (ISCO) Study and Intrusive Drum Removal/Landfill Cap (2009). Sampling responsibilities included coordinating sampling efforts for several sites within the project area, soil sampling, water sampling, petroleum, oil and lubricant (POL) sampling and packing/shipping of sampling. Tasks included report writing and gathering field supplies.
 - Environmental Scientist for EPA 1004 Former Skelly Site Assessment, Winnebago, Nebraska (October 2008). The project consisted of conducting a site assessment at a potential LUST site on the Winnebago Reservation in Nebraska, following NDEQ guidelines for a Tier 1 Site Assessment. Tasks included writing the Site Health and Safety Plan, installing soil borings, monitoring wells and collecting soil and groundwater samples.

- Environmental Scientist for Choggiung East Creek Hatchery Post Treatment sampling and assessment report, Dillingham, Alaska (October 2008). Duties included developing sampling grid, soil sampling, collecting field-screening headspace samples, using a photoionization detector (PID), and packing and shipping of samples. Wrote a report summarizing field activities, presenting analytical data, and providing recommendations for future site remediation.
- Environmental Scientist for Private Residence Heating Fuel Investigation, Dillingham, Alaska (October 2008). Developed a sampling protocol and performed soil sampling of an excavation at a private residence in Dillingham, Alaska. Duties included developing sampling grid, soil sampling, and packing and shipping of samples.
- Environmental Scientist, providing project support for Elmendorf Treatability Study, Elmendorf Air Force Base, Alaska (June 2008). Provided assistance for installation of bladder pump and set up of micro purge system for groundwater sampling from monitoring wells. Calibrated YSI brand water quality meter and logging system for groundwater monitoring. Performed seep sampling using a peristaltic pump. Assisted in labeling, packing and shipping of samples.
- Environmental Scientist for FAA Cape Yakataga Landfill Removal project, Phase III, Cape Yakataga, Alaska (May 2008). Collected waste characterization and confirmation soil samples for the decommissioning of a landfill and Biocell.
- Environmental Scientist acting as the coordinator for manifesting barge shipments of contaminated soil to a disposal facility.
- Environmental Scientist for Annette Island Phase I Environmental Due Diligence Audit (EDDA) (April 2008). Project responsibilities included conducting site visits to check for environmental contamination, interviews, database searches, and preparation of report and figures.
- Environmental Scientist for Private Housing development project, Anchorage Alaska, (April 2008). Performed on-site assistance for well placement for groundwater contamination study.
- Environmental Scientist for USACE, Beaufort Sea project, North Slope, Alaska, (August 2007). Performed remedial investigation sampling at Kogru, Collinson Point, and Nuvagapak DEW Line sites. Assisted in following work plan, sampling soil, sediment and surface water samples, sample packing, and shipping.
- Environmental Scientist for Bristol Environmental & Engineering Services Corporation (insert start date? - March 31, 2008).
- Environmental Scientist for Alaska Natural Gas Development Authority (ANGDA) Wetland Delineation, various locations, Alaska (Summer 2008). Performed wetland delineation on sections of an approximately 470-mile proposed natural gas pipeline corridor. The effort was initiated by ANGDA to prepare primary requirements for a U.S. Army Corps of Engineers (USACE) National Environmental Policy Act (NEPA) ecological evaluation. Duties included traversing through developed and undeveloped Alaska wilderness, navigation and data entry using ArcPad software on several models of Trimble GPS units, making determinations of whether areas along the route were wetlands or uplands, participating in all aspects of wetland delineation, including digging pits, identifying soil types using Munsell soil charts, and identifying local plant types. Training included wildlife health and safety, wildlife interaction, rare plant Identification, wetland procedures, and using Geographical Information Systems to prepare a Wetland Delineation Report, which included: Wetland and

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Waterways Report, Preliminary Project Description, Support Data (Field forms, JD Forms, Photographs) and Mapping.

- Staff Biologist for Bering Sea Fishermen's Association, Anchorage, Alaska (2003 to March 2007). Developed fisheries research project in rural western Alaska and interior Alaska. Aided in the facilitation of these fisheries projects, as well as provided on-site guidance and hands-on research. Developed and maintained strong relationships with State fish and game entities. Developed contracts and research plans for fisheries research. Conducted data collection and storage. Acted as support staff of the Arctic-Yukon-Kuskokwim Sustainable Salmon Initiative. Planned data sharing symposiums and meetings. Provided oversight for many aspects of several fisheries projects. Maintained frequent contact with state, federal, and non-governmental employees for field projects. Performed grant writing and contract development. Responsible for maintaining ongoing compliance with grant criteria. Participated in watershed council meetings, resource advisory committees, Alaska Board of Fisheries Meetings, North Pacific Fisheries Management Council meetings, and various other fisheries-related meetings. Assisted Executive Director and Program Director with fisheries issues as they arose. Performed operations in remote areas, including field camp setup and maintenance, weir installation, and project preparation, setup, and maintenance. Traveled extensively to projects across the state of Alaska.
- Fisheries Technician II for the Alaska Department of Fish and Game (2001 to 2003). Worked on the Yukon River, Kuskokwim River, and several other Western Alaska and Interior Alaska rivers, as well as Bristol Bay. Traveled to and lived in remote areas and performed camp setup. Performed radio tagging salmonids. Used gill netting as a capture method. Performed scale taking, scale reading, tissue sampling, and otolith extraction on herring. Performed Age-Sex-Length (ASL) sampling. Performed river navigation and utilized Global Positioning System. Maintained fish wheels as a means of data collection and used data loggers. Identified salmon and resident species.
- Lab Aide for Eastern Washington University, Cheney, Washington (1998 to 1999). Collected walleye ASL information. Read walleye scales. Assisted in separating out juvenile preserved fish by species. Performed backpack and boat electrofishing and collected samples from an electrofishing boat. Assisted in collecting individual and population statistics.

Software

Microsoft Word and Excel ArcPad Software gINT Geotechnical and Geoenvironmental Software

Training and Certifications

HAZWOPER 40-hour Training CPR and First Aid for Adults Certified Erosion and Sediment Control Lead Wetland Training Institute Wetland Delineation Certification Program Defensive Driving Training

Affiliations

American Fisheries Society (Non-active)

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LYNDSEY KLEPPIN

Geologist

Areas of Expertise

- ✓ Environmental Sampling
- ✓ Risk Assessment
- ✓ Geologic Research
- ✓ Borehole Geophysical Logging

Ms. Kleppin is a geologist with experience in exploration geology, geophysical investigation, and contaminated sites projects. She is proficient in producing geologic maps, well diagrams, cross sections and reports. Ms. Kleppin has several years of experience in the environmental field performing surface water, groundwater, soil and sediment sampling, as well as administrative and technical support, field logistics, instrumentation, risk assessment, and technical writing.

Education

B.A., Geology, Carleton College, Northfield, Minnesota 2004

Project Experience

- Geologist for Bristol Environmental Remediation, LLC (March 31, 2008 present).
 - Project support for U.S. Environmental Protection Agency (EPA) contract to investigate and remediate leaking underground storage tank (LUST) sites on Indian Lands (2010). Conducted Idaho Department of Environmental Quality Risk Evaluation Phase 2 for a petroleum-impacted site. Authored technical memorandum presenting RE-2 results recommending risk-based site closure.
 - Field Lead for Native American Lands Environmental Mitigation (NALEMP) Site Assessment in Tetlin, Alaska (2009). NALEMP was developed by the Department of Defense (DoD) to address environmental issues from past DoD activities on Indian lands Conducted preliminary site assessment and prepared the Strategic Project Implementation Plan (SPIP) for submittal to the USACE.
 - Field Team Leader for Fort Richardson Monitoring Well Inventory Project (2009). Conducted background research and field investigations of 250+ points using a Trimble GPS unit to create a comprehensive monitoring well database for USACE. The database included determination of active/inactive status based on sampling event records and location within active operable units or POL release sites.
 - Environmental Scientist for Fort Wainwright Operating Unit 3 (2009). Collected low-flow groundwater samples for DRO, GRO, VOC, EDB, PAH, iron (II), lead, and sulfate analysis.
 - Geologist for U.S. Environmental Protection Agency (EPA) contract to investigate and remediate leaking underground storage tank (LUST) sites on Indian Lands (2009). Created soil boring logs and collected analytical soil samples. Generated lithologic cross sections and well diagrams for Region 9 Navajo sites using gINT Geotechnical software and produced technical memos reporting groundwater monitoring events.
 - Geologist for Bristol Bay Native Corporation Land Department (2009). Researched and prepared historical and geologic background summary for use by mineral appraiser in region of prospective land exchange area.
 - Field team leader for mineral exploration project downhole survey program (2008).

- Managed borehole geophysical logging program for Cu-Au-Mo Prospect near Iliamna, Alaska. Developed site-based Standard Operating Procedure for ABI Acoustic Televiewer, 2PCA-100 Caliper, Full Wave Sonic Sonde and 4WNA Winch. Revised procedures to optimize data quality and downhole tool recovery. Performed maintenance and repairs on equipment. Trained operators and provided regular reports to site staff.
- Geologist for Bristol Environmental & Engineering Services Corporation (May 2007 March 2008).
 - Field Geologist for Pebble Project (2007). Conducted borehole geophysical surveys with and provided general field support for Northern Dynasty's Pebble Cu-Au-Mo prospect near Iliamna, Alaska. Additional activities included surface water and soil sampling, ground topographic surveying, corelogging, geotechnical logging, and logistical support.

Professional Experience

- Weekend Programs Lead Teacher for the Pacific Science Center in Seattle, Washington (2006 to 2007). Taught interactive science lessons and assisted in curriculum development.
- Assistant to the Director for Osservatorio Geologico di Coldigioco in Italy (2005 to 2006). Provided winter logistics and maintenance for geologic observatory.
- Adjunct Chemistry Instructor for University of Alaska Anchorage (2005). Duties included laboratory instruction, as well as creating and grading chemistry exams.
- Field Studies Instructor for 3D Education and Adventure, Isle of Wight, England (2004). Activity and field studies instructor at outdoor education camp for schoolchildren.
- Prudhoe Bay summer hire for NANA Corporation, Prudhoe Bay, Alaska (2001). Seasonal laborer at field camp facility in the Prudhoe Bay oilfield.

Software

Microsoft Office Suite including Word, Excel and Access

Matrix Geotechnical Acquisition Software

ArcPad Software

gINT Geotechnical and Geoenvironmental Software

Training and Certifications

40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER)

8-Hour HAZWOPER Refresher

Hazardous Materials Transportation, (DOT/IATA) 49 CFR 172.700-704 and Section 1.5 IATA Compliance

Alaska Department of Environmental Conservation 8-Hour Soil Vapor Intrusion Course

CPR and First Aid with current 4-Hour Refresher

Rigging and Slinging

APPENDIX B

TestAmerica Laboratory Certifications (Provided on CD)

APPENDIX C

Field Screening Laboratory Standard Operating Procedures

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ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
AAC	Alaska Administrative Code
ADEC	Alaska Department of Environmental Conservation
AK102	Alaska Method determination of DRO
AK103	Alaska Method determination RRO
ASE	accelerated solvent extractor
CVS	CalibraTion Verification Standard
DCS	diesel calibration standard
DE	Diatomaceous Earth
DRO	diesel range organics
FID	flame-ionization detector
GC	gas chromatographic or gas chromatograph
ICAL	initial calibration
LCS	laboratory control sample
LFB	laboratory-fortified blank
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligram per liter
mL	microliter
MSDS	Material Safety Data Sheet
NOM	naturally occurring materials
OTP	ortho-terphenyl
PQLS	practical quantitation limitS
psi	pounds per square inch
QC	quality control
RCS	residual calibration standard
RRO	residual range organics (motor oil range)
RSD	relative standard deviation
RTW	retention time window
SOP	Standard Operation Procedure
VOA	volatile organic analysis

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March 2010

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedures for determining the concentration of diesel range organics and residual range organics (DRO/RRO) in soil using methodology developed by the Alaska Department of Environmental Conservation (ADEC), and described in the *Underground Storage Tank Procedures Manual* (ADEC, 2002).

1.1 **OBJECTIVES**

The objectives in the use of this method are to accurately determine the concentrations of diesel and residual range organics in soil.

1.1.1 Scope of Method

These methods are designed to measure the concentration of DRO and RRO in soil. DRO is determined by method AK102, and RRO is determined by method AK103. The diesel range corresponds to an n-Alkane range from the beginning of C₁₀ to the beginning of C₂₅, and a boiling point range of approximately 170 degrees Celsius (°C) to 400 °C. An n-Alkane is a chemical compound that consists of only hydrogen and carbon, linked in a single bond in a straight chain. The residual range corresponds to an n-alkane range from the beginning of C₂₅ to the end of C₃₆, and a boiling range of 400 °C to 500 °C. Both methods are performed sequentially on a single sample extract, and a single analytical run on a gas chromatograph. The methods differ in the range of quantitation, based on the elution of n-alkanes on the gas chromatographic (GC) column.

1.1.2 Practical Quantitation Limits

The practical quantitation limits (PQLs) for these methods have been adjusted to reflect sitespecific cleanup levels. The PQLs for DRO and RRO have been elevated to approximately 500 milligrams per kilogram (mg/kg).

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1.1.3 Dynamic Range

The dynamic range for method AK 102 is 500 milligrams per liter (mg/L) to 25,000 mg/L. The dynamic range for method AK 103 is 500 mg/L to 25,000 mg/L. The dynamic ranges reflect the concentration of target analytes in the sample extract. Dilutions may be performed as necessary to put the chromatographic envelope (sample extract concentration) within the linear range of the method. The determination of soil concentrations is based on the sample weight and the percent moisture in the sample (Sections 9.12.1 and 9.12.2).

2.0 METHOD SUMMARY

2.1 METHOD PROCEDURE

This method provides GC conditions for the detection of semivolatile petroleum products, such as diesel and motor oil. Other non-petroleum compounds with similar characteristics and boiling points may also be detected with this method.

Samples are extracted from approximately 20 grams of soil using methylene chloride as the solvent. A surrogate mixture of known concentration is spiked into all field and quality control (QC) samples to evaluate the efficiency of the extraction process. An aliquot (2 micro liters $[\mu L]$) of the extract is injected into a gas chromatograph equipped with a capillary column and a flame ionization detector (FID). The GC is temperature programmed to facilitate separation of organic compounds.

2.1.1 DRO Range

Quantitation of DRO is performed by comparing the total chromatographic area between and including the peak start of C_{10} to the peak start of C_{25} , including both resolved and unresolved compounds, based on the FID response compared to a diesel calibration standard. Integration is performed using forced baseline-baseline integration.

2.1.2 RRO Range

Quantitation of RRO is performed by comparing the total chromatographic area between and including the peak start of C_{25} to the peak end of C_{36} , including both resolved and unresolved components. Integration is performed using forced baseline-baseline integration.

2.2 METHOD DEVELOPMENT

This method was developed by the ADEC and is based, in part, on a modification of the American Petroleum Institute consensus "Method for the Determination of Diesel Range Organics," Revision 2, 2/5/92, supplemented with information gathered by the State of Alaska, Department of Environmental Conservation, State Chemistry Laboratory, with support from the Storage Tank Program. It is also based in part on EPA Methods 8000 and 8100, SW – 846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* [1], adopted by reference in Title 18 Alaska Administrative Code, Chapter 78.090(i) [18 AAC

Alaska Methods AK102 and AK103

78.090(i)], Method OA-2 [2] and work by the EPA Total Petroleum Hydrocarbons Method Committee [3], and the State of Oregon, "Total Petroleum Hydrocarbon Methods" QAR 340-122-350, dated December 11, 1990.

3.0 DEFINITIONS

3.1 DIESEL RANGE ORGANICS (DRO)

All chromatographic peaks for DRO, both resolved and unresolved, eluting between the peak start of n-decane (C_{10}) and the peak start of n-pentacosane (C_{25}). Quantitation is based on direct comparison of the area within this range to the total area over the same (C_{10} - C_{25}) range of the calibration standard, as determined by FID response using forced baseline-baseline integration. Surrogate peak areas shall be determined by valley to valley integration.

3.2 **RESIDUAL RANGE ORGANICS (RRO)**

All chromatographic peaks for RRO, both resolved and unresolved, eluting between the peak start of n-pentacosane (C_{25}) and the peak end of n-hextriacontane (C_{36}). Quantitation is based on direct comparison of the area within this range to the total area over the same ($C_{25} - C_{36}$) range of the calibration standard, as determined by FID response using forced baseline-baseline integration. Surrogate peak areas shall be determined by valley-to-valley integration.

3.3 DIESEL CALIBRATION STANDARD (DCS)

The DCS is Commercial #2 diesel fuel or equivalent hydrocarbon mixture, in which greater than 95% of the hydrocarbon mass elutes within the diesel change and is diluted to appropriate concentrations in methylene chloride. The DCS serves as a calibration standard for DRO. The DCS standard will be injected without any other standards present to demonstrate the 95% elution criteria is met.

3.4 **RESIDUALS CALIBRATION STANDARD (RCS)**

RCS is an equal blend of 30 weight and 40 weight motor oils (1:1), diluted to appropriate concentrations in methylene chloride. The RCS serves as a calibration standard for RRO. The RCS standard will be injected without any other standards present to demonstrate the elution range of the RCS.

3.5 COMBINED CALIBRATION STANDARD

A stock standard mixture of DCS and RCS components is used for the initial and continuing calibration standards. Multiple concentrations of the combined calibration standards are used

for the initial calibration. The standard concentrations vary from the PQL of 500 mg/L to 25,000 mg/L, which is the upper dynamic range of the calibrations. A 5,000 mg/L standard is used as the continuing calibration standard.

3.6 CONTINUING CALIBRATION STANDARD (CCS)

The continuing calibration standard is a mid-range working standard diluted from the stock standard solution and is used to verify that the analytical system is responding in a manner comparable to the time of initial calibration. The continuing calibration standard is analyzed at the beginning of an analytical sequence, and after every 20 samples to ensure that reported sample concentrations are accurate, as determined by the calibration.

3.7 CALIBRATION VERIFICATION STANDARD (CVS)

The CVS is a QC standard, but with diesel from a source other than that used to prepare the DCS, (i.e., a second source). It is used by the laboratory to verify the accuracy of calibration and source materials. Greater than 95 % of the hydrocarbon mass must elute within the diesel range, as described in Section 3.1.

3.8 SURROGATE MIXTURES

Ortho-terphenyl is used as the DRO surrogate and n-triacontane d^{62} is used as the RRO surrogate. The surrogate mixture contains equal concentrations of the surrogates, and it is spiked into all extracted samples before the extraction begins.

3.9 RETENTION TIME WINDOW (RTW) STANDARD

The RTW is a mixture of the normal (n-) alkanes, including n-decane, n-pentacosane, and n-hexatriacontane (C₁₀, C₂₅ and C₃₆), which are analyzed once every 24-hour day or with each analytical batch of samples. This standard defines the integration windows for methods AK102 and AK103.

3.10 STANDARD SOIL

Baked Ottawa sand is used in QC samples (method blank and laboratory-fortified blank) to represent the soil matrix. Quality control samples are extracted and analyzed using the same procedures as field samples.

3.11 METHOD BLANK

The method blank (also known as a procedural blank), demonstrates that the apparatus and reagents used to verify that the handling, extraction, and analysis of field samples is valid and that the reported concentrations in field samples were not biased due to contamination introduced in the extraction and analysis process.

3.12 INSTRUMENT BLANK

An instrument blank demonstrates that the instrument is free from contamination. The instrument blank is not extracted, and consists of methylene chloride solvent used in the extraction process.

3.13 SOLVENT BLANK

A solvent blank demonstrates that the solvent (in this case methylene chloride) used in the method is free from contamination. It may also serve as an instrument blank.

3.14 LABORATORY-FORTIFIED BLANK (LFB)

An LFB is a method blank sample spiked with diluted Commercial #2 diesel fuel and motor oil which is the same as that used to make the Combined Calibration Standard (see Section 3.5 of this method). There are 2 laboratory-fortified blanks extracted with every extraction batch. The spike recoveries are used to evaluate method control for accuracy and precision (see Table 1 of this method in Section 11.2). The laboratory-fortified blank is synonymous with a laboratory control sample (LCS).

3.15 METHOD DETECTION LIMIT (MDL)

The MDL is the minimum concentration of a compound that can be measured and reported with 99% confidence that the value is greater than zero, determined from analysis of a sample in a given matrix containing the analyte(s). The MDL is determined prior to the analysis of any samples.

3.16 PRACTICAL QUANTITATION LIMIT (PQL)

The PQL is defined as the concentration in the sample extract that can be accurately determined and has a reproducible result. The PQL is generally between 2 and 5 times the MDL.

4.0 INTERFERENCES

4.1 NON-TARGET ANALYTES

Other organic compounds, including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters, and biogenic compounds, are measurable under the conditions of this method.

4.2 **BIOGENIC INTERFERENCE**

Some site conditions contain non-petroleum compounds from naturally occurring materials (NOMs), such as plants. Many of these compounds found in natural settings also occur at varying concentrations in crude oil and refined petroleum products. When NOM is present in a DRO or RRO sample, there is no practical method to distinguish NOMs from petrogenic sources. This interference is termed biogenic interference. Silica gel may be used to remove some of the polar compounds and reduce the magnitude of quantitative interference to varying degrees. Sample chromatograms of refined products usually have a distinct characteristic hump, or bell shape. Chromatograms from NOM samples do not exhibit the bell shape and typically have a ramped look that extends from the middle diesel range past the residual range. The analysts experience will be used for the interpretation of chromatograms when the presence of NOM is suspected. Silica gel may be employed to lessen the magnitude of interference.

4.3 GLASSWARE CLEANING

Method interferences are reduced by washing all glassware with hot soapy water, followed by a rinse with tap water and methylene chloride At least one blank must be analyzed with each extraction batch to demonstrate that the laboratory samples are free from method interferences.

4.4 REAGENT QUALITY

High purity reagents must be used to minimize interference problems. All reagents are screened for contamination before being introduced to field and QC samples.

Alaska Methods AK102 and AK103

4.5 SAMPLE CARRYOVER

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, the successive analysis will be evaluated for possible carryover.

4.6 WATER

Water may be unintentially extracted along with the target analytes during the extraction process, particularly when samples are wet. Water interferes with the proper concentration of the extract, and also interferes with the analysis. The water must be removed using steps outlined in Section 9.2.1.5.

5.0 SAFETY ISSUES

5.1 CHEMICAL EXPOSURE

The toxicity or carcinogenicity of each reagent in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of Material Safety Data Sheets will be maintained on site, and made available to all personnel involved in chemical analysis.

5.2 HEARING PROTECTION

Hearing protection will be used when performing sonication.

5.3 SAMPLE DRYING

The ADEC requires that moisture determinations must accompany all soils data (reported in mg/dry kg) in order to determine the results in the original soil condition. Because of the potential for high petroleum compound concentrations in the soil, all drying should be done under a functioning hood.

(Intentionally blank)

6.0 APPARATUS AND MATERIALS

6.1 GLASSWARE

- 4-oz amber glass wide-mouth jars with Teflon[®]-lined screw caps
- 60-mL volatile organic analysis (VOA) vials with Teflon-lined screw caps are used as collection vessels for the accelerated solvent extractor (ASE)
- Two mL glass vials with Teflon-lined cap (autosampler vials)
- Disposable pipettes: Pasteur
- Graduated cylinders: 250-mL
- Glass funnels
- Volumetric flasks: 10-mL, 25-mL, 50-mL, 250-mL, and 1000-mL
- Micro syringes 1-μL, 5-μL, 10-μL, 25-μL, 100-μL, and 500-μL.

6.2 ANALYTICAL BALANCE

An analytical balance capable of accurately weighing to 0.0001 grain will be used for preparing standards. A top-loading balance capable of weighing to the nearest 0.01 grain will be used for sample preparation and percent moisture determination.

6.3 SONICATION

6.3.1 Ultrasonic Cell Disrupter (Sonicator)

A horn-type sonicator equipped with a titanium tip (Heat Systems-Ultrasonics, Inc., Model W-400 (475 watt)) with pulsing capability and a No. 200, ½-inch tapped disrupter horn is used to perform extraction method 3550B.

6.3.2 Sonabox

The sonicator will be operated in a sonabox to decrease sound. Hearing protection will also be worn by lab personnel during sonication steps to prevent hearing loss.

6.4 ACCELERATED SOLVENT EXTRACTOR

A Dionex ASE[®] 200 (Accelerated solvent extractor ASE), is used to perform EPA extraction method 3545. The ASE employs pressurized solvent and heat to increase the extraction efficiency of samples.

Alaska Methods AK102 and AK103

6.5 SOLVENT CONCENTRATOR

A solvent evaporator (TurboVap[®]) with a nitrogen gas source will be used to concentrate sample extracts to their final volume.

6.6 MISCELLANEOUS APPARATUS

- Stainless steel spatula.
- Weigh boats
- Glass wool

6.7 GAS CHROMATOGRAPH (GC)

A GC is an analytical system that measures concentrations of analytes introduced with an autosampler and syringes into an injection port. The components in the sample extract separate inside of a 30-meter analytical column before their response is measured on an FID. A data system capable of measuring peak areas using a forced baseline-baseline projection is required. The data system is capable of storing and processing chromatographic data.

6.7.1 Columns

Columns are Restek DB-5 30 M x 0.53 mm 1.0 micron film thickness or equivalent.

6.7.1.1 Optional Columns

Other columns may be used as long as they are capable of achieving the necessary resolution. The column must resolve C₁₀ from the solvent front in a mid-range DCS or CVS.

7.0 REAGENTS AND STANDARDS

7.1 REAGENT WATER

Reagent water is free of organics, target analytes, and interfering substances.

7.2 METHYLENE CHLORIDE

Methylene chloride – reagent grade or equivalent. At a minimum, the solvent must be shown to be free of DRO, as demonstrated by the analysis of a solvent blank.

7.3 SODIUM SULFATE

Sodium sulfate – (ACS grade) granular, anhydrous. Sodium sulfate is used to remove water from samples in extraction method 3550B. Water interferes with the extraction and concentration of sample extracts. Sodium sulfate is purified by heating it in a shallow tray at 400 °C for 4 hours in a muffle furnace. Incomplete cleaning of sodium sulfate can result in DRO contamination of samples. Refer to Section 4.0 for other interferences

Note: Sodium sulfate should not be used with samples that will be extracted with the ASE.

7.4 DIATOMACEOUS EARTH

Diatomaceous Earth (DE) is used to dry samples for extraction method 3545. DE is purified by heating it in a shallow tray at 400 °C for 4 hours in a muffle furnace. Incomplete cleaning of DE can result in DRO contamination of samples.

7.5 STOCK STANDARD SOLUTIONS

Stock Standard Solutions for AK102 and AK103 analyses are prepared in methylene chloride. Standard preparation will follow the procedures as described in Section 9.1. All standards prepared by the laboratory must be stored at less than 6 °C, and protected from light. The meniscus is marked and observed to ensure stock standard integrity. Standards must be replaced within 6 months of preparation. Prepared standards purchased from commercial suppliers may be kept indefinitely, and under the conditions, specified by the manufacturer if different than described in this paragraph. Stock standards often come in flame-sealed glass ampoules, and with proper storage are good for one year from receipt.

7.5.1 Surrogates

A Surrogate Control Standard is a working standard of 1 μ g/mL each of OTP and hexatriacontane-d⁶² in methylene chloride is used as a working standard solution. When PQLs are elevated due to a lower concentration of extracts, the surrogate concentration or volume added to samples must be adjusted accordingly. A calculated volume of concentrated stock solution may be combined with initial and continuing calibration standards to verify that surrogate recoveries and chromatographic separation are adequate for the determination of extraction recovery efficiencies.

7.5.2 Diesel and Residual Range Calibration Standards

Diesel #2 is used to prepare stock calibration standards in methylene chloride. No fewer than 5 concentrations of this DCS are used for instrument calibration. Other than one standard concentration near the PQL, the expected range of concentrations found in project samples should define the working range of the GC.

7.5.2.1 Continuing Calibration Standard

A mid-range dilution of the diesel range and residual range blends serve as the Continuing Calibration Standard. The concentration is approximately 10,000 mg/L.

7.5.3 RTW Standard

A Retention Time Window Standard is a stock solution containing at a minimum, n-alkanes C_{10} , C_{25} and C_{36} each, at a level of at least 2 µg/mL. This blend of alkanes serves as a RTW, defining the mix for DRO and RRO.

7.5.4 Stock Calibration Verification Standard (CVS)

The CVS is prepared from a second source of commercial Diesel #2 other than that used to prepare the DCS, as described in Section 7.5.2 of this method. A working solution is made at a recommended concentration of 5000 μ g/mL in methylene chloride, which is near the midpoint of the calibration range.

8.0 SAMPLE COLLECTION, PRESERVATION, CONTAINERS, AND HOLDING TIMES

8.1 SAMPLE COLLECTION

Soils for field analyses may be collected in labeled Ziploc[®] bags or 4-oz amber glass jars with Teflon-lined lid. A separate Sampling and Analysis Plan or Field Sampling Plan should fully address the procedures used to collect field samples. Samples must be collected using clean sampling equipment, and new clean nitrile gloves must be worn. Sample gloves should be changed prior to the beginning of any collection activities and between samples.

8.2 SAMPLE PRESERVATION

All samples will be immediately placed in an iced cooler after collection, and stored at 4 ± 2 °C until extraction.

8.3 HOLDING TIMES

Sample extraction must be performed within 14 days [1]. All analyses of extracts must take place within 40 days.

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9.0 **PROCEDURE**

9.1 STANDARDS PREPARATION

9.1.1 Initial and Continuing Calibration Standards and Surrogates

DRO calibration standards are prepared from neat #2 Diesel. RRO standards are prepared from equal portions of 30-weight and 40-weight motor oil. Neat standards are weighed on a 4-place analytical balance. Approximately 2.5 grams of #2 Diesel and 2.5 grams of the mixed motor oils are added to a 100-mL volumetric flask. Methylene chloride is added to the volumetric flask to a final volume of 100 mL, generating a combined stock standard solution at a concentration of 25,000 mg/L. Other initial and continuing calibration standards are prepared from this stock standard solution.

Initial and continuing calibration standards are prepared by diluting the stock standard solution in volumetric flasks on a volume:volume basis. Initial calibration standards are prepared at concentrations of 500, 2500, 5000, and 10,000 mg/L. The stock standard solution is used for the 25,000 mg/L solution, which is the upper dynamic range of the calibrations.

The 5,000 mg/L solution is used at the continuing calibration standard.

Ortho-terphenyl and n-triacontane-d62 are added to the stock calibration standard at 10 mg/L from a vendor-prepared solution (Ultra Scientific). Subsequent dilutions of the stock standard will result in surrogate concentrations of 0.2, 1, 2, and 4 mg/L.

9.2 ACCELERATED SOLVENT EXTRACTION

Method 3545A (ASE) is used for soil samples and the extraction solvent is methylene chloride.

9.2.1 Soil Preparation – Accelerated Solvent Extraction

The following sections outline procedures used to prepare sample extracts for analysis.

9.2.1.1 <u>Remove Excessive Water</u>

Decant any water layer that may accompany the solid layer in the sample. Note the apparent condition of the sample (presence of foreign materials, variable particle size, presence of oil sheen, multiple phases, etc., on the bench sheet).

9.2.1.2 Sample Weighing

Weigh 10 grams to 30 grams of the original sample in a tared weighing dish on a 2-place balance. Add an equal weight of DE, and stir the mixture well with a clean stainless steel or Teflon spatula. The sample should have a grainy texture after mixing. If the sample clumps, add more DE until a grainy texture is achieved, and note the addition. (Do this for all samples and standards.)

9.2.1.3 Sample Transfer and Spiking

Place the soil-DE mixtures into the ASE 33-mL extraction tubes, and add surrogate to both field and QC samples. Prepare the method blank and LFBs in a similar fashion to field samples. Add a known amount of spiking solution to the duplicate LFBs. These QC samples should contain 20 grams of Ottawa sand and an equal amount of DE.

9.2.1.4 ASE Program

Place the extraction tubes on the ASE and select Program 5 (DRO-RRO extraction). Be sure the solvent bottles are full with clean methylene chloride, and that rinse reservoirs are empty and in place.

9.2.1.5 Sample Extract Drying

If water was unintentially transferred to the extract, the extract may need to be dried by passing the extract through a clean filter funnel, lined with glass wool and topped with anyhydrous sodium sulfate. The eluent can be collected directly into a TurboVap tube during the drying step. This step ensures that the extract is water-free before concentration.

9.2.1.6 Sample Extract Concentration

Transfer the extract into a clean concentration vessel (TurboVap tube), and concentrate extracts to 20 mL at a temperature not to exceed 55 °C. Transfer an aliquot of extract to 2

labeled GC vials for analysis. Extracts should be stored at <6 $^{\circ}$ C. Record the information for the extraction and concentration steps.

9.3 SONICATION EXTRACTION

9.3.1.1 <u>Remove Excessive Water</u>

Decant any water layer that may accompany the solid layer in the sample. Note the apparent condition of the sample (presence of foreign materials, variable particle size, presence of oil sheen, multiple phases, etc., on the bench sheet).

9.3.1.2 Sample Weighing

Weigh 10 grams to 30 grams of the original sample in a tared weighing dish, or 250-mL beaker on a 2-place balance. Add an equal weight of DE or sodium sulfate, and stir the mixture well with a clean stainless steel or Teflon spatula. The sample should have a grainy texture after mixing. If the sample clumps, add more DE or sodium sulfate until a grainy texture is achieved and note the addition. (Do this for all samples and standards.)

9.3.1.3 Sample Transfer and Spiking

Place the dried soil mixture into a 250-mL beaker and add surrogate to both field and QC samples. Prepare the method blank and LFBs in a similar fashion to field samples. Add a known amount of spiking solution to the duplicate LFBs. These QC samples should contain 20 grams of Ottawa sand.

9.3.1.4 Sonication

Add approximately 50 mL of methylene chloride to the sample after surrogate has been added. Place the beaker under the sonicator and sonicate for 90 seconds. Transfer the solvent extract to a TurboVap tube that has a lined glass filter funnel filled with sodium sulfate. Repeat sonication twice more by adding 50 mL of solvent each time.

9.4 SAMPLE CONCENTRATION

Samples must be concentrated to a measurable final volume of 20 mL, using a TurboVap. TurboVap tubes are placed in the TurboVap, and solvents are evaporated under a gentle

nitrogen stream in a heated water bath. Samples must not go dry, or the extraction process will need to be repeated with fresh soil.

9.5 MOISTURE DETERMINATION FOR SOLIDS

9.5.1.1 Wet Sample Weight

To determine percentage of moisture, pre-weigh an aluminum drying pan and record the weight to the nearest 0.00 grams. Tare the balance to zero with the aluminum pan on the balance and weigh 5 -10 grams of the sample in the pan. Record the weight to the nearest 0.01 gram. Dry the sample a minimum of 4 hours or overnight in a warm, 105 °C oven.

9.5.1.2 Dry Sample Weight

Remove the sample from the oven and cool until the sample reaches room temperature, these weigh to the nearest 0.01 gram. Record the weight.

9.5.2 Percent Moisture Calculation

Subtract the aluminum boat weight from the dry weight and divide the result by the wet weight. Multiply the result by 100% to determine the percent dry weight. The wet weight is equal to 1.0 minus the dry weight, expressed as a decimal.

9.6 SAMPLE EXTRACT DILUTION TECHNIQUE

Measure 1.0 mL of sample into a 10-mL volumetric flask. Dilute sample to 10-mL with methylene chloride. Transfer to a labeled vial with a Teflon-lined lid. Note the dilution on the vial. Mark meniscus and store at <4 °C.

9.7 GAS CHROMATOGRAPHY

9.7.1 Method Conditions

Set helium column pressure to 20 pounds per square inch (psi). Set oven temperature to 40 °C for 2 minutes, then ramp at a rate of 15 °C/minute to 320 °C, and hold for 12 minutes (run time = 30.6 minutes). Set FID to 320 °C and injector to 280 °C. Method conditions may be modified to achieve proper separation of analytes. The instrument must be calibrated after any method conditions have changed.

9.7.2 Method Performance Criteria

GC run conditions and columns must be chosen to meet the following criteria:

- Resolution of the methylene chloride solvent front from C₁₀.
- The column must be capable of separating typical diesel and residual components from the surrogates. There may be potential problems with separating the resolution of $n-C_{19}$ from OTP and $n-C_{21}$ at varying relative concentrations.

9.8 CALIBRATION

9.8.1 Initial Calibration

To calibrate the GC, set up as in Section 9.2 of this method. A minimum of five concentrations of DCS must be used for the calibration. The lowest initial calibration standard concentration will establish the PQL for the method, and the highest concentration standard defines the upper quantitation limit. Samples exceeding the upper calibration limit must be diluted and reanalyzed.

9.8.2 Initial Calibration Curve Verification

The calibration curve must be confirmed using the CVS. This standard independently verifies the accuracy of the calibration. The concentration of the CVS should be within the expected concentration range of the samples to be analyzed. A relative standard deviation (RSD) of less than 20% of true value is the acceptance criteria for the CVS.

9.8.3 Continuing Calibration Standards (CCS)

The working calibration curve must be verified on each working day (24 hours) by the injection of a continuing calibration standard (see Section 3.6 of this method) at a concentration near the mid-point of the calibration curve. The continuing calibration standard is a diluted aliquot of the same standard used to initially calibrate the instrument. An initial calibration standard near the mid-point of the curve may be used for the continuing calibration standard varies from the predicted response by more than 25%, a new calibration curve must be prepared. The instrument should be checked and cleaned prior to establishing a new 5-point calibration.

9.8.4 Calibration Curve Linearity

Acceptable criteria for the initial calibration is dependent on the type of curve fit applied to the initial calibration. Acceptance criteria for the most used types of calibration curves are listed below.

- A linear regression curve fit must have an R² of 0.995 or better,
- A quadratic fit must have an R² of 0.995 or better,
- Average of response factors, the average percent relative standard deviation (%RSD) is less than 20% over the working range.
- Other curve fits may be employed as long as they meet acceptance criteria outlined in EPA method 8000B [2].

9.9 ESTABLISHING RTWS

9.9.1 RTW Definition

The RTW for individual peaks is defined as the average RT plus or minus three times the standard deviation of the absolute retention times for each component. The RTWs for this method are defined in Section 3.9. RTWs are crucial to the identification of target compounds. RTWs are established to compensate for minor shifts in absolute retention times as a result of sampling loadings and normal chromatographic variability.

9.9.2 Chromatographic Separation Definition

Chromatographic processes achieve separation by passing a mobile phase over a stationary phase. Constituents in a mixture are separated because they partition differently between the mobile and stationary phases, and thus have different retention times. Compounds that strongly interact with the stationary phase elute slowly (i.e., long RTs), while compounds that remain in the mobile phase with little interaction with the stationary phase elute quickly (short RTWs).

Before establishing RTWs, be certain that the GC system is within optimum operating conditions (Section 6.7). Make three injections of the RTW Standard (Section 7.5.3) and surrogates (Section 7.5.1) throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in RTWs that are too tight.

9.9.3 Calculation of RTWs

- 1. Record the retention times for for decane, pentacosane, and hexatriacontane using an RTW standard (Section 7.5.3) and the surrogates (Section 7.5.5.) from at least 3 injections over a minimum 72-hour period.
- 2. Calculate the mean and standard deviation of the three absolute retention times for the RTW standards and surrogates.
- 3. In those cases where the standard deviation for a particular analyte is zero, the laboratory will use ± 0.05 minute as the default standard.
- 4. The width of the RTW for each analyte, surrogate, and major constituent is multicomponent analytes is defined as ± 3 times the standard deviation of the mean absolute RT established during the 72-hour period. If the default standard deviation in Step 3 is used, the width of the window will be 0.03 minutes.

9.9.4 Reestablishing RTWs

The laboratory must calculate RTWs for each standard on each GC column, and whenever a new GC column is installed or instrument conditions change. The data must be retained by the laboratory for at least a year. RTWs must be verified regularly and updated no less frequently than once a year.

9.10 GAS CHROMATOGRAPH ANALYSIS

9.10.1 Injection Volume

Samples are analyzed by GC/FID. Injection volumes are 2 μ L, using the conditions established in Section 9.7 of this method.

9.10.2 Analytical Batch Window

If initial calibration (Section 9.8.1) has been performed, verify the calibration by analysis of a mid-point continuing calibration standard. An analytical batch is defined as the analysis of standards, field samples, and QC samples analyzed sequentially until all samples are analyzed, or those samples analyzed within 24 hours.

9.10.3 Continuing Calibration Acceptance Criteria

Calculate the percent difference of the response from the known continuing calibration standard concentration and the established response factor in mg/L. If the reported continuing

calibration standard has a reported concentration difference greater than 25% from the known concentration, corrective action must be taken.

9.10.4 Instrument Blank Criteria

The instrument blank is essential for determining if analytical conditions are suitable for the proper analysis of samples. An unextracted solvent blank (methylene chloride) is analyzed each day to determine the area generated from normal baseline noise under the conditions prevailing in the 24-hour period. This area is generated by projecting a horizontal baseline between the retention times observed for the peak start of C₁₀ and the peak start of C₂₅. This blank is integrated over the DRO area in the same manner as for the field samples, and is reported as the solvent blank. Baseline subtractions of instrument blanks is not allowed.

9.10.5 Carryover Blanks

Blanks may be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination above the PQL, maintenance must be performed to remove the source of the carryover before any samples can be analyzed. New injector liners may be installed, or the column may be trimmed or baked out to remove the chromatographic contamination. Subsequent blanks must be analyzed until the system is shown to retain contaminant at concentrations less than the one-half the PQL.

9.10.6 Calibration Exceedences

If the DRO concentration exceeds the linear range of the method (as defined by the range of the calibration curve) in the final extract, corrective action must be taken. The sample should be diluted and the response of the major peaks should be kept in the upper half of the linear range of the calibration curve.

9.11 CHROMATOGRAPHIC INTERPRETATION

The analyst may perform a qualitative interpretation of sample chromatograms in order to determine if the sample result is attributed to natural (anthropogenic) or petroleum (petrogenic) sources. Chromatograms from known types of petroleum products may be used to compare the fuel patterns to those found in samples. Field notes and sample examination may also be used to identify potential origins of analytes in the chromatograms.

9.12 CALCULATIONS

9.12.1 Percent Moisture Calculation for Soils

% Moisture = $[(A-C)/(A-B)] \times 100$

Where:

A = weight of boat + wet sam	ple
------------------------------	-----

B = weight of boat

C = weight of boat + dry sample

Note: Make sure drying oven is placed under a hood. Heavily contaminated soils will produce strong organic vapors.

9.12.2 Dry Weight Calculation for Soil

mg/dry kg soil = (100-% moisture)/100)) x wet weight of sample

The % moisture calculation will be included in the data package.

Note: Excel spreadsheets with formulas will be used to determine the percent moisture, dry weight of samples, and soil sample concentrations.

9.12.3 Soil Concentration Calculation

External Sample Calculation:

Soil samples:

$$Cs = \underline{Cex * (Vt) * D}$$
(Ws)

Where:

*	=	times
Cs	=	Concentration of DRO or RRO in mg/kg
Cex	=	Concentration in final extract
Vt	=	Volume of final extract in mL
D	=	Dilution factor, if dilution was performed on the sample prior to analysis.
		If no dilution was made, then $D = 1$, dimensionless

Ws = Dry weight of sample extracted in grams

9.12.4 Data Reduction Software

A software program from Agilent (Chemstation-Enviroquant) will be used to perform calculations relative to Sections 9.12 of this method, with minimal analyst intervention.

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10.0 QUALITY CONTROL

10.1 CURVE VERIFICATION STANDARD (CVS)

- The CVS is not extracted.
- The CVS is analyzed once after the initial calibration standards to verify calibration curve.
- The CVS recovery limit is 75-125% of true value.

10.2 CONTINUING CALIBRATION SAMPLES

- The continuing calibration standard is not extracted.
- The continuing calibration standard is analyzed at the start and end of an analytical batch, and for every 20 samples in that batch.
- The continuing calibration standard recovery requirement is 75-125% of true value.

10.3 BLANKS

- The instrument blank may be analyzed with each analytical batch to demonstrate that the system is free from contamination.
- The method blank must be extracted and analyzed with each extraction batch.
- If additional cleanup steps are performed on field samples, the same steps must be applied to the method blank.
- Acceptance Criteria: Results for the method blank must be less than or equal to the reporting limit concentration, or less than 5% of the lowest concentration found in the associated samples.
- BLANK SUBTRACTION IS NOT ALLOWED. Blanks are reported by value.
- Other blanks may be analyzed as necessary following the recommendations of Chapter 2, Section 9 of the UST Procedures Manual.

10.4 LABORATORY FORTIFIED BLANKS (LFB)

- LFB is extracted using the method procedure.
- Two LFBs are analyzed with each extraction batch.
- Acceptance Criteria: The LFB recovery requirement for AK102-DRO is 75-125% of true value. The LFB recovery requirement for AK103-RRO is 60-120%. The RPD is 20% for both methods.
- If additional cleanup steps are performed on field samples, the same steps must be applied to the LFB samples.
- If any LFB recovery fails to meet method criteria, appropriate corrective action must be taken. See Section 10.6 Corrective Actions.

10.5 SURROGATES

10.5.1 Surrogate Concentration

The surrogate should be spiked at a level to produce a recommended extract concentration of $1.66 \ \mu g/mL$.

10.5.2 Surrogate Acceptance Criteria

Surrogate recoveries must be 60-120% for LCS (continuing calibration standard, CVS, method blank, LFB), and 50-150 % for field samples (all other samples).

10.5.3 Surrogate Recovery Failure-Corrective Action

If any surrogate recovery fails to meet method criteria, corrective action must be taken if there is no reasonable explanation for the failed recovery. Some soil types such at peat and tundra often bias recoveries low. See Section 10.6 Corrective Actions.

10.5.4 Sample Qualifiers (Flags)

If field samples show poor surrogate recovery that is not attributable to laboratory error, DRO results must be flagged.

10.6 CORRECTIVE ACTIONS

The actions listed below are recommended and may not apply to a particular failure.

- If the CVS fails to meet acceptance criteria, recheck all calculations used to prepare the standards. If the CVS fails again, prepare new ICAL and CVS standards from neat standards.
- If the instrument fails to meet continuing calibration criteria, all samples analyzed since the last acceptable continuing calibration standard must be reanalyzed.
- If method blank acceptance criteria are not met, identify and correct the source of contamination and re-prepare and reanalyze the associated samples.
- If the LFB(s) acceptance limits are not met, reanalyze the LFB to confirm the original result is reliable. If the results are still outside control limits, the associated samples must be re-extracted and reanalyzed. If the LFB is above the upper control limit, and the associated samples are all below the PQL, the deviation should be described in a non-conformance memo.

Alaska Methods AK102 and AK103

• If surrogate recoveries are outside the established limits, verify calculations, dilutions, and standard solutions. Also, verify that instrument performance is acceptable. High recoveries may be due to co-eluting matrix interference, and the chromatogram should be examined for evidence of this. Low recoveries may be due to adsorption by the sample matrix (clay, peat, or organic material in the sample). Recalculate the results and/or reanalyze the extract if the checks reveal a problem. If the surrogate recovery is outside of established limits due to well-documented matrix effects, the results must be flagged.

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March 2010

11.0 METHOD PERFORMANCE

11.1 METHOD DETECTION LIMIT

The MDL for soil is calculated according to Title 40 Code of Federal Regulations, Part 136 (40 CFR136), Appendix B (1994). The MDL is estimated to be 60 mg/kg (external standard calibration, Ottawa sand) for DRO and 89 mg/kg for RRO. MDL studies will be performed and MDLs will be updated prior to any sample analyses.

11.2 METHOD ACCEPTANCE CRITERIA FOR AK102

The method acceptance criteria for laboratory control and field samples analyzed by Method AK102 are presented in Table 1.

	Control Limits		
Soils (mg/kg)	% Recovery	Relative % Difference	
Laboratory-Fortified Blanks	75-125	20	
Continuing Calibration	75-125		
Calibration Verification	75-125		
Surrogate Recovery:			
Laboratory Control Samples**	60-120		
Field Sample	50-150		

Table 1 Method AK102 Acceptance Criteria for Quality Control

Notes:

**Laboratory Control Sample is any laboratory prepared sample used for quality control, except for calibration standards. Field criteria from voluntary contribution of method performance information from approved laboratories, and method performance at SCL.

% = percent

mg/kg = milligrams per kilogram

11.3 METHOD ACCEPTANCE CRITERIA FOR AK103

The method acceptance criteria for laboratory control and field samples analyzed by Method

AK103 are presented in Table 2.

Table 2 Method AK103 Acceptance Criteria for Quality Control

	Control Limits		
Soils (mg/kg)	% Recovery	Relative % Difference 20	
Laboratory Fortified Blanks	60-120		
Continuing Calibration	75-125		
Calibration Verification	75-125		
Surrogate Recovery:			
Laboratory Control Samples**	60-120		
Field Sample	50-150		

**Laboratory Control Sample is any laboratory prepared sample used for quality control, except for calibration standards. Field criteria from voluntary contribution of method performance information from approved laboratories, and method performance at SCL.

% = percent

mg/kg = milligrams per kilogram

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- U.S. Environmental Protection Agency. SW 846-Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Solid Waste Method 8000B, Determinative Chromatographic Separations. Revision 2, 1996. Washington, D.C.

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Field Screening for Polychlorinated Biphenyls (PCBs) by Gas Chromatography

STANDARD OPERATING PROCEDURE

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STANDARD OPERATING PROCEDURE

Field Screening for Polychlorinated Biphenyls (PCBs) by Gas Chromatography

ACRONYMS AND ABBREVIATIONS

v

µg/L	micrograms per liter
ANSI	American National Standards Institute
ECD	electron capture detectors
EPA	U.S. Environmental Protection Agency
GC	gas chromatograph (or gas chromatogram)
LCS	laboratory control sample
LFB	laboratory-fortified blank
MDL	method detection limit
mg/kg	milligrams per kilogram
mL	milliliter
MSDS	Material Safety Data Sheet
NOM	natural organic matter
PCBs	polychlorinated biphenyls
PE	performance evaluation
PIDs	photoionization detectors
PPE	personal protective equipment
ppm	parts per million
QC	quality control
RF	response factor
RSD	relative standard deviation
SOP	Standard Operating Procedure
TCMX	tetrachlorometaxylene
TSDF	treatment storage disposal facility
VOA	volatile organic analysis

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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedures for determining the concentration of polychlorinated biphenyls (PCBs) as Aroclors[®] using the methodology developed by U.S. Environmental Protection Agency (EPA) Region 1 and described in the *Standard Operating Procedure PCB Field Testing for Soil and Sediment Samples* (EPA 2002).

1.1 PURPOSE OF METHOD

This method may be used to determine the concentrations of PCBs as Aroclors in extracts from soil and solids using open-tubular, capillary columns with electron capture detectors (ECD). The Aroclors listed below have been determined by this method, using a singlecolumn analysis system. This method also may be applied to other matrices, such as oils and wipe samples, if appropriate sample extraction procedures are employed.

Aroclor Class	CAS Registry No.ª
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

Ta	ble	1	Aroclor®	Classes
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Notes:

^aChemical Abstract Service Registry No.

1.2 AROCLOR QUANTITATION

The seven classes of Aroclors listed in Table 1 are those that are commonly specified in EPA regulations. The quantitation of PCBs as Aroclors is appropriate for meeting standard State and EPA cleanup criteria.

1.3 AROCLOR IDENTIFICATION

Compound identification based on single-column analysis is appropriate when Aroclor patterns of known standards (fingerprints) can be compared to a sample chromatogram. Certified standards of the differing Aroclors are used to produce chromatograms, which can be compared to sample chromatograms to identify the Aroclor mixture so it can be properly quantitated. Software which incorporates chromatogram overlay tools or other means may also be used to compare chromatograms of unknown mixtures against standards. The overlay tool is especially useful in determining if weathering of the Aroclor has occurred.

1.4 AROCLOR MIXTURES

Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analytical expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns compared to those of Aroclor standards.

2.0 SUMMARY OF METHOD

2.1 EXTRACTION

Approximately 10 grams of soil (wet weight) is weighed in a tared sample boat on a 2-place, top-loading balance, and the weight is recorded on a spreadsheet. Approximately 10 grams of the same sample is weighed in a tared aluminum drying pan for percent moisture determination. Approximately 5 grams of dried sodium sulfate is added to the soil to remove the water in the soil, more sodium sulfate may be added if the sample is still "wet". The sample is transferred to a 40 milliliter (mL) volatile organic analysis (VOA) vial then 1 mL of 2 milligrams per liter (mg/L) parts per million (ppm) of surrogate is added to the sample using a gas-tight syringe. Ten mL of a 1:1 hexane acetone mixture is then added to the VOA vial and sealed with a Teflon[®] cap. The contents of the vial are agitated for 1 minute using a vortex mixer or vigorous shaking by hand. Two mL of deionized water is added to the vial to facilitate the separation of hexane from acetone in the vial. The vial contents are briefly hand mixed and allowed to settle. Separation and settling may be assisted by placing the vial in a hand-cranked centrifuge (Whirlybird) and spinning the vial(s) for 30 seconds. The hexane and all analytes of interest are contained in the top-floating layer in the vial. If the sample extract shows signs of petroleum contamination, acid cleanup may be performed to remove interferents. Approximately 4 mL of the hexane layer is transferred to two 2mL crimp top vials. The sample extract is now ready for analysis.

2.2 ALTERNATE EXTRACTION METHODS

Solid samples may be extracted with hexane-acetone (1:1) using Method 3545A (2007a) (pressurized fluid extraction) or Method 3550C (2007b) (ultrasonic extraction), or other appropriate technique or solvents. Extraction methods are presented in Section 10.1.

2.3 EXTRACT CLEANUP

Extracts for PCB analysis may be subjected to a sulfuric acid cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides, as well as petroleum. Therefore, this method is not applicable to the analysis of organochlorinated compounds, such as pesticides.

2.4 SAMPLE INJECTION

After cleanup, the extract is analyzed by injecting a 2-microliter (μ L) aliquot into a gas chromatograph (GC), equipped with a wide-bore fused-silica capillary column and an electron capture detector (ECD).

2.5 SAMPLE QUANTITATION

Sample quantitation involves two distinct steps. First the Aroclor chromatographic pattern has to be qualitatively identified against a known standard (fingerprinting). Second, the five major quantitative peaks must be integrated using consistent integration technique in order to properly quantitate the concentration of Aroclor in the extract. Each peak is quantified separately, and the determined concentrations of each of the 5 peaks are added to determine to total PCB concentration in the extract. The soil concentration is calculated using the soil dry weight, final volume of the extract (hexane layer), and any dilutions performed on the final extract. Sample results are reported in milligrams per kilogram (mg/kg) on a dry weight basis.

3.0 **DEFINITIONS**

The following sections provide definitions that may be relevant to this procedure, but may not include all terms used in this method.

3.1 POLYCHLORINATED BIPHENYLS (PCBs)

PCBs are a class of chlorinated organic compounds with 1 to 10 chlorine atoms attached to the biphenyl rings. There are 209 possible compounds (congeners) of PCBs. Each congener contains varying levels of chlorine ions attached to the carbon atoms of 2 conjoined phenyl rings. The manufacturing of the PCBs produced 7 main classes of PCBs, known as Aroclors. The 7 main classes of Aroclors are listed in Table 1 in Section 1.1.

3.2 INTEGRATION

Integration is the determination of the area of a peak or peaks in a chromatogram. Integration determines the base or bottom of the peak, and it separates the integrated peak from other peaks. Software generally performs the integration automatically; however, the analyst may be required to manually integrate the peak. The peak integration must be consistent with the integration performed on the initial and continuing calibration standards. Proper integration is required for accurate quantitation.

3.2.1 Quantitation

Quantitation is the determination of standard and sample concentrations based on the instrument response to known standard concentrations. Quantitation is based on the ratio of response (area) to concentration, and the ratio is known as the calibration or response factor.

3.2.2 Extraction

Extraction is the transfer of analytes from the matrix (soil) into solvent (extract) for the determination of analyte concentrations in the matrix.

3.2.3 Elution

Elution is the transmittal of separated analytes from the GC column to the detector.

3.2.4 Combined Calibration Standard

A stock standard mixture of Aroclor 1016 and Aroclor 1260 is diluted in hexane to produce the initial and continuing calibration standards. Multiple concentration standards are used for the initial calibration and the standard concentrations vary from the practical quantitation limit (PQL) of 0.1 to 40 mg/L, which is the upper dynamic range of the initial calibration. A 1.0 mg/L standard is used as the continuing calibration standard.

3.2.5 Continuing Calibration Standard (CCS)

A mid-range working standard diluted from the Stock Standard Solution, used to verify that the analytical system is responding in a manner comparable to that at the time of initial calibration. The continuing calibration standard is analyzed at the beginning of an analytical sequence, and at minimum, after every 20 samples to ensure that reported sample concentrations are accurate as determined by the initial calibration.

3.2.6 Calibration Verification Standard (CVS)

The CVS is a quality control (QC) standard, prepared as outlined in Section 8.6 of this method, but with an Aroclor mixture from a source other than that used to prepare the Initial Calibration, i.e., a second source from a different vendor. It is used by the laboratory to verify the accuracy of calibration and standards. Acceptance criteria are +/- 20% of the initial calibration response factor.

3.2.7 Surrogate Mixture

Tetrachlorometaxylene (TCMX) and decachlorobiphenyl are used as the surrogates for this method. The surrogate mixture contains equal concentrations of the surrogates, and it is spiked into all extracted samples before the extraction begins. The surrogate mixture is also included in the initial calibration standard as varying concentrations. Decachlorobiphenyl is the primary surrogate used to evaluate the extraction efficiency. Tetrachlorometaxylene is the secondary surrogate standard and may be used to evaluate the extraction efficiency when decachlorobiphenyl is subject to interference, as described in Section 4.2.

3.2.8 Standard Soil

Baked Ottawa sand is used in QC samples (method blank and laboratory-fortified blanks) to represent the soil matrix. Quality control samples are extracted and analyzed using the same procedures as field samples.

3.2.9 Method Blank

Method blank, also known as a preparation blank, demonstrates that the apparatus and reagents used to verify that the handling, extraction, and analysis of field samples are valid, and that the reported concentrations in field samples were not biased due to contamination introduced in the extraction and analysis process.

3.2.10 Instrument Blank

Instrument blank demonstrates that the instrument is free from contamination. The instrument blank is not extracted and consists of hexane.

3.2.11 Solvent Blank

A solvent blank demonstrates that the solvent (in this case hexane) used in the method is free from contamination. It may also serve as an instrument blank.

3.2.12 Laboratory-Fortified Blank (LFB)

A method blank sample consisting of Ottawa sand is spiked with a known quantity of prepared standard that is the same as that used to make the Initial and Continuing Calibration Standards (see Section 3.2.4 and 3.2.5 of this method). Two LFBs are extracted with every extraction batch. The spike recoveries are used to evaluate method control for accuracy and precision (see Table 1 in Section 1.1 of this method). The LFB is synonymous with a laboratory control sample (LCS).

3.2.13 Method Detection Limit (MDL)

The MDL is the minimal concentration of a compound that can be measured and reported with 99% confidence that the value is greater than zero, determined from analysis of a sample in a given matrix containing the analyte(s). (See, Appendix B, for the method of determining MDL). The method detection limit is determined prior to the analysis of any field samples.

3.2.14 Practical Quantitation Limit (PQL)

The PQL is defined as the concentration in the sample extract that can be accurately determined, and has a reproducible result. The PQL is generally between 2 and 5 times the MDL.

3.2.15 Extraction Batch

An extraction batch is a set of field and QC samples extracted using the same consistent procedure throughout the batch. A sample batch consists of an extraction blank, two LFBs, and up to 20 field samples extracted in less than a 24 hour period.

4.0 INTERFERENCES

4.1 SOLVENTS, REAGENTS, GLASSWARE

Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and solvents may be necessary. Refer to each method to be used for specific guidance on QC procedures, and to Section 6.4.1 for general guidance on the cleaning of glassware.

4.2 DECACHLOROBIPHENYL

Decachlorobiphenyl is used as a surrogate, but it may also be present as an analyte of interest when the PCB analyte is Aroclor 1268. Aroclor 1268 is not a major class of PCBs, and it was rarely used in practice. In this instance, dechlorobiphenyl is a target analyte, but the chromatographic result should not be used to determine surrogate recovery nor for quantitation of the Aroclor. Instead, TCMX should be used to measure recovery efficiency as a surrogate, and another major chromatographic peak should be used to quantitate the Aroclor against known calibration standards.

4.3 INTERFERENCES FROM PHTHALATES

Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations.

Exhaustive cleanup of solvents, reagents, and glassware may be required to eliminate background phthalate ester contamination.

These materials can be removed prior to analysis using EPA Method 3665 (sulfuric acid cleanup).

Cross-contamination of clean glassware can routinely occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

4.4 SULFUR (S_8)

Sulfur (S₈) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur contamination should be expected with sediment samples. Sulfur can be removed through the use of EPA Method 3665.

4.5 PETROLEUM

Petroleum may be extracted from samples as a non-target analyte. Petroleum interferes with the quantitation of PCBs when it co-elutes with the PCBs. Petroleum can be removed from samples following a sulfuric acid cleanup (EPA Method 3665) of the extract.

4.6 **OTHER INTERFERENCES**

Interferences extracted from the samples will vary considerably from matrix to matrix and sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories, as follows:

- Contaminated solvents, reagents, or sample processing hardware.
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
- Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) may cause interference of some of the Aroclor peaks.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment, and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of Material Safety Data Sheets (MSDSs) will be maintained and will be available to all personnel involved in these analyses.

5.1 SAFETY REQUIREMENTS

5.1.1 Personal Protective Equipment (PPE)

Eye protection that satisfies ANSI Z87.1 specifications (splash-proof and shatter-proof eye protection), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been removed are discarded as nonhazardous waste. Non-disposable gloves must be cleaned immediately.

5.1.2 High Temperature Surfaces

The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations in those zones, and must cool them to room temperature prior to working on them. Solid reagents, such as silica gel, Ottawa Sand, and diatomaceous earth, are baked in a muffle furnace at high temperatures (450°C). Care must be taken when placing solid reagents in the muffle furnace and removing them after heating. It is required that commercial-grade oven mitts and tongs are used for the muffle furnace. The soil-drying oven is used to remove water from soil samples in order to determine the percent moisture in samples. Oven mitts must be used when placing or removing samples from the oven.

5.1.3 Electrical Hazards

There are areas of high voltage in the GC. Depending on the work to be performed, either turn off the power to the instrument, or unplug the GC from the power source. It should be noted that the back of the GC has capacitors that store energy even if the GC is unplugged. Avoid contacting the capacitor. If working in the capacitor area, it is required that the analyst wears a grounding strap.

5.1.4 Radiation

The ECD contains radioactive nickel (⁶³Ni) that requires leak testing every six months. The detector can be maintained without risk to the operator as long as the source is left in its sealed vessel. Do not open up the source, it is in violation of licensing agreements with Agilent Technologies and the nuclear regulatory commission. If a source leak is suspected, do not use the detector. Perform a wipe test to evaluate the potential leak and contact Agilent immediately for further instructions. A leaking source cannot be transported by air, unless it is in a container made specifically for shipping radioactive items. Proper documentation and manifesting is required. A non-leaking detector can be flown on aircraft as hazardous material in excepted quantities. The contained radiation of a single detector is 15 millicuries.

5.1.5 Solvent Handling

Solvents used for sample extraction may be flammable and/or hazardous. Personnel must minimize their exposure to solvent fumes and avoid contact with skin or clothing. Refer to each MSDS to properly identify hazards associated with each type of solvent. Eye protection is required when handling solvents. Solvents must be handled under a fume hood whenever they are transferred. Residual solvent may remain in soil after extraction, and the soil must be stored under a fume hood or in a proper container after extraction. Signs of solvent exposure include dizziness, coughing, lightheadedness, and headaches. Over exposure to hexane may cause irritation to the skin and eyes. Hexane and acetone are flammable and must be handled with care under a fume hood. Sulfuric acid is a corrosive material, and will produce chemical burns when exposed to the skin. Sulfuric acid must be handled under a fume hood. Sulfuric acid vapors are an irritant and may cause problems with the respiratory tract and mucous membranes. Organic vapor monitors (PIDs) and/or chemical badges may be worn to ensure exposure levels are minimized.

5.1.6 Target Analytes

Some target analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Standard materials and stock standard solutions of these compounds and field samples should be handled with suitable protection to the skin, eyes, etc.

6.0 EQUIPMENT AND SUPPLIES

Glassware, reagents, supplies, equipment, and settings other than those listed in this procedure may be employed provided that method performance is appropriate and not impacted by the use of items not listed in this method.

6.1 GAS CHROMATOGRAPH

An analytical system complete with GC suitable for split-splitless injection and all necessary accessories, including auto-injectors, syringes, analytical columns, gases, ECDs, and a data system.

6.2 GC COLUMNS

The single-column approach will be utilized and involves a single analysis to determine if PCBs are present. The chromatographic pattern will confirm the identity of the compound. The single-column approach may employ narrow-bore (0.25 or 0.32-mm ID) or wide-bore (0.53-mm ID) columns. The GC may employ dual columns mounted in a single GC, but with each column connected to a separate injector and a separate detector.

The columns listed in this section may be used at the discretion of the analyst performing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available.

- 30-m DB-5 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.0-μm film thickness.
- 30-m DB-608 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5-µm or 0.83-µm film thickness.
- 30-m DB-1701 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

6.3 ANALYTICAL BALANCES

- An analytical balance capable of weighing to 0.0001 gram balance is used for the preparation of standards.
- A 2-place, top-loading balance capable of weighing to 0.01 gram is used for the determination of sample weights for extraction and percent moisture determinations.

• Calibration weights will accompany the balances, and the balance calibration and accuracy are checked daily prior to sample or standard weighing.

6.4 GLASSWARE

- 4-oz amber glass wide-mouth jars with Teflon-lined screw caps
- 40-mL VOA vials with Teflon-lined screw caps are used as extraction vessels
- Two mL glass vials with Teflon-lined crimp caps (autosampler vials)
- Transfer pipettes
- Graded pipettes are pipettes with volumes etched on the glass of such quality to accurately measure the volume contained in the pipette
- Glass Beakers: 250-mL
- Glass funnels
- 10-mL, 25-mL, and 50-mL volumetric glass used for the preparation of standards.

6.4.1 Glassware Cleaning

Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing (Alconox[®]) with hot water, and rinsed with tap water and/or organic-free reagent water. Glassware should be covered with aluminum foil and stored in a clean environment between uses.

6.5 EXTRACTION EQUIPMENT

- Vortex Shaker
- Heat Systems Model W400 Ultrasonic Extractor with ¹/₂" horn
- Dionex Model ASE 200 Accelerated Solvent Extractor

6.6 OTHER EQUIPMENT

- GOW-MAC[®] Model 21-250 helium leak detector. The leak detector is used to verify system integrity by checking all fittings and orifices for leaks that could affect system performance.
- Glass wool

7.0 REAGENTS AND STANDARDS

Reagent-grade or pesticide-grade chemicals are used in all preparations and extractions. Other grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

NIST-certified standards will be used for the identification and quantitation of target analytes.

7.1 SOLVENTS

Solvents used in the extraction and cleanup procedures include *n*-hexane, acetone, sulfuric acid, and water. All solvents must be exchanged to *n*-hexane prior to analysis. All solvents are pesticide grade in quality or equivalent, and each lot of solvent must be determined to be free of phthalates. A manufacturer's certificate of analysis is sufficient determination, unless factors or interferences indicate otherwise.

Hexane is used for the preparation of all standards except for surrogates and spiking solutions. All surrogates and spiking solutions will be in acetone. All solvent lots must be reagent- or pesticide-grade in quality, or equivalent, and should be determined to be free of phthalates.

7.2 ORGANIC-FREE REAGENT WATER

All references to water in this method refer to organic-free reagent water

7.3 STANDARD SOLUTIONS

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000B for additional information on the preparation of calibration standards.

7.4 STOCK STANDARD SOLUTIONS

Stock standard solutions (1,000 μ g/mL) of certified PCB standards in acetone are purchased from vendors such as Restek or AccuStandard. Certificates of analysis are maintained and

stored on site in order to ensure the accuracy of prepared standards. Lot numbers and each standard preparation are recorded in the Standards Log Book.

NOTE: Standard solutions (stock, composite, calibration, and surrogate) are stored at less than 6°C in Teflon-sealed glass containers in the dark once they are removed from flame-sealed vials. When a lot of standards are prepared, aliquots of that lot are stored in individual small vials. All stock and working standard solutions must be replaced after six months, or sooner if routine QC checks indicate a problem.

7.5 CALIBRATION STANDARDS FOR AROCLORS

7.5.1 Initial Calibration Standard Mixtures

A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 are used to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

A minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 are prepared by diluting a stock standard with hexane. The concentrations should correspond to the expected range of concentrations found in real samples, and must be within the linear range of the detector. Initial calibration standards are prepared in volumetric glassware at concentrations of 0.1, 0.5, 1.0, 10 and 40 mg/L from a 1000 mg/L stock standard solution. Other concentrations may be used as long as they demonstrate response and linearity consistent with other standards, and are within the linear dynamic range of the detector.

7.5.2 Single PCB Standards

Single standards of each of the other five Aroclors listed in Table 1 are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in

Section 7.5.1 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors listed in Table 1 also may be used to determine the calibration factor for each Aroclor when a linear calibration model is chosen. A standard for each of the other Aroclors is prepared at a concentration of 1.0 mg/L. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed at the discretion of the analyst based on project requirements.

7.5.3 Surrogate Standards

The performance of the method is monitored using surrogates. Surrogate standards (TCMX) are added to all samples, method blanks, laboratory-fortified blanks, and calibration standards. The surrogate spiking solutions used on samples are prepared in acetone.

7.5.4 Other Standards

Other standards (e.g., other Aroclors) and other calibration approaches (e.g., non-linear calibration for individual Aroclors) may be employed to meet project needs. When the nature of the PCB contamination is already known, standards of those particular Aroclors will be used to prepare initial and continuing calibration standards.

STANDARD OPERATING PROCEDURE

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(Intentionally blank)

February 2010

8.0 QUALITY CONTROL

The QC acceptance criteria for various aspects of this method are described in this section.

Quality control limits are outlined in Table 2 and described in detail in the following sections.

QC Item	Frequency	Acceptance Criteria	Corrective Action(s)
Initial Calibration	Before analysis of samples	<20% RPD or a linear regression correlation coefficient (r ²) value greater than 0.995	Check standard integrity and perform additional initial calibrations as necessary.
Continuing Calibration	Before introduction of samples, after every 10 samples, and at the end of an analytical batch	<20% RPD of the known standard concentration	Inject another standard, clean the injector port. Perform initial calibration.
Instrument Blank	Before introduction of samples, after every 10 samples, and at the end of an analytical batch	Reported concentrations less than ½ the practical quantitation limit	Repeat blank injection, clean injection port, and replace septa and liner.
Extraction Blank	One extraction blank is extracted and analyzed with each extraction batch.	Reported concentrations less than ½ the practical quantitation limit	Repeat blank injection, clean injection port, and replace septa and liner. If the blank concentration is less than 10 times the lowest concentration of any field samples, data must be qualified (flagged) or the entire sample batch must be re- extracted.
Laboratory- Fortified Blank (LFB)	Two LFBs are extracted and analyzed with each extraction batch.	Control limits are 60 to 130% of known spiked concentrations. The RPD between 2 LFBs from the same extraction batch must not exceed 30%.	Repeat injection, if re-injection fails to meet acceptance criteria, all samples in the extraction batch must be re- extracted.
Surrogates	Surrogates are included in all continuing calibration standards, method blanks, LFBs and field samples.	continuing calibration standard acceptance criteria are +/- 20% RPD of the known concentration. Method blanks and LFB acceptance criteria are 40-140% for TCMX and 60-130% for DCB.	Determine the cause of the failure. Failure to meet recovery criteria in method blanks and LFBs indicate that extraction or analysis problems exist. Failure of surrogate recoveries in field samples may indicate matrix interference if recoveries are acceptable in extraction blanks and LFBs.

Table 2	Quality	Control	Criteria
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QC Item	Frequency	Acceptance Criteria	Corrective Action(s)		
Performance Evaluation (PE) Samples	Once a year or when the laboratory or instrumentation has gone through significant changes	Within the manufacturer's acceptance criteria	Determine to cause of failure, make necessary changes and repeat analysis with a new PE Sample.		
Notes: CCS = con	tinuing calibration standard				

Table 2 Quality Control Criteria (continued)

CCS = continuing calibration standard QC = quality control RPD = relative percent difference RSD = relative standard deviation

8.1 SAMPLE COLLECTION

The collection of analytical field samples is described in the Sample Analysis Plan, which is a separate document. The Sample Analysis Plan translates project objectives and specifications into procedures used in the collection of samples. Samples must be collected using clean sampling equipment, and new clean nitrile gloves must be worn. Sample gloves should be changed prior to the beginning of any collection activities and between samples.

8.2 INITIAL CALIBRATION

The initial calibration is performed by analyzing standards at known variable concentrations over the expected concentration range of samples, or within the linear dynamic range of the detector. The area (response) of quantitative peaks is determined, and then the area is divided by the known concentration to develop individual response factors. The response factors may be incorporated into a calibration function, such as an average response factor or a linear regression. An average response factor incorporates the individual response factors into an average of the response factors. The average response must have a relative standard deviation (RSD) of less than 20% to be acceptable. A linear regression calibration curve uses the least squares method to produce a straight line that does not pass through the origin, when the regression calibration technique is used. The linear regression must have a correlation coefficient (r^2) greater than 0.995 to be acceptable. The software (Agilent ChemStation and Enviroquant) performs the calculations necessary to determine the average RSD and correlation coefficient (r^2).

8.3 CONTINUING CALIBRATION

A continuing calibration standard is analyzed as a calibration check, after each group of 20 samples in the analysis sequence. Thus, injections of method blank and LFB extracts and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, are also not counted in the total. The response factors for the calibration must be within ± 20 percent of the initial calibration to meet acceptance criteria. When the continuing calibration is outside of acceptance criteria, the laboratory will stop analyses and take corrective action.

8.4 LABORATORY FORTIFIED BLANK (LFB)

The LFB concentration of PCBs as Aroclor 1260 is spiked at sufficient volume to have the concentration at 1.0 mg/L in the blank sample. When specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for the LFB. Other concentrations may be used, as appropriate for the intended application. The LFB is also known as the LCS. Two LFBs are extracted with each extraction batch.

8.5 METHOD BLANK

Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank is prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the RTW of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, before processing the samples, if possible. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory must monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. A single method blank is extracted with each extraction batch.

8.6 SAMPLE QUALITY CONTROL FOR PREPARATION AND ANALYSIS

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). This includes the analysis of QC samples, including a method blank and LFBs in each analytical batch and the addition of surrogates to each field sample QC sample when surrogates are used. Any method blanks, matrix spike samples, or replicate samples, should be subjected to the same analytical procedures (Section 11.0) as those used on actual samples.

8.7 SURROGATE RECOVERIES

The laboratory will evaluate surrogate recovery data from individual samples versus the surrogate control limits listed in Table 2.

8.8 INITIAL DEMONSTRATION OF PROFICIENCY (PERFORMANCE EVALUATION [PE] SAMPLE)

Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained, or significant changes in instrumentation are made. PE samples are provided by manufacturers at concentrations unknown to the laboratory or analyst. Once the PE sample concentration is determined, the results are sent back to the manufacturer for confirmation. If the confirmation is within the manufacturer's criteria, a certificate of performance is issued by the manufacturer. If the confirmation result is outside of acceptance criteria, the cause(s) must be corrected before a new PE sample is requested. The analysis and determination of each PE sample, whether in or out of acceptance criteria, must be documented and maintained by the laboratory.

9.0 METHOD PROCEDURES

The following procedures have been demonstrated to be applicable for soil screening by the Office of Environmental Measurement and Evaluation (EPA Region 1). The method is also described in *Standard Operating Procedures for PCB Field Testing For Soil and Sediment Samples* (EPA, 2002).

9.1 SAMPLE EXTRACTION

Soil Samples are extracted by weighing approximately 10 grams (wet weight) of sample in a weigh boat. Sodium sulfate is added to the soil to remove water. The weighed sample is transferred to 40-mL VOA vial, then surrogates are introduced to the sample. Ten (10) mL of 1:1 hexane-acetone solvent is added to the sample and agitated with a vortex mixer for 90 seconds. 2 mL of organic-free water is added to separate the hexane from the acetone and the sample is again agitated on the vortex shaker for 30 seconds. The extraction vial is then centrifuged with a hand-cranked centrifuge (Whirlybird) for 30 seconds or more to facilitate the separation of the hexane from the soil and acetone-water layer. The hexane layer is the top layer, and it is removed and transferred to two 2 mL autosampler vials for analysis.

EPA Method 3550C, ultrasonic extraction, or EPA Method 3545A, accelerated solvent extraction (with a Dionex ASE 200), may be used to handle large sample loads, difficult matrices, or, in the event of mechanical breakdown, poor recoveries. A sample batch will only be extracted using one method.

The use of hexane-acetone solvents generally reduces the amount of interferences, and improves signal-to-noise ratio.

9.1.1 Extract Cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples may require additional preparation to remove interferences before analysis. A modified Method 3665A will be used for PCB sample cleanup when sample extracts exhibit likely non-target interference due to the presence of POL or natural organic matter (NOM). The hexane layer is removed from the top of the sample extract after water has been added to facilitate the separation of the hexane and

acetone. Target analytes preferentially partition into the hexane layer. The hexane layer is removed and transferred to a clean 40-mL VOA vial using transfer pipettes. Five mL of 1:1 sulfuric-acid-water is then applied to sample extract, mixed on a vortex shaker, and allowed to settle before injection on the GC.

9.1.2 Method Applicability to Other Matrices

The extraction techniques for solids may be applicable to wipe samples and other sample matrices not addressed in Section 10.1. The analysis of oil samples may need special sample preparation procedures that are not described here.

9.1.3 Demonstration of Extraction Method Proficiency and Detection Limits

Reference materials, field-contaminated samples, and spiked samples will be used to verify the applicability of the selected extraction techniques. Samples will be spiked with the compounds of interest and surrogates in order to determine the percent recovery and the limit of detection for each extraction method.

A combination of Aroclor 1016 and Aroclor 1260 will be spiked at concentrations at or below the PQL to determine the detection limit. The PQL has been empirically determined to be 0.1 mg/kg in soil samples.

9.2 GC CONDITIONS

9.2.1 Single-Column Analysis

This capillary GC/ECD method allows the analyst the option of using 0.25-mm or 0.32-mm ID capillary columns (narrow-bore), or 0.53-mm ID capillary columns (wide-bore). Due to the likely presence of non-target interference, 0.53-mm ID columns will be used for this analysis. The GC is configured with dual injectors, dual columns, and dual detectors for simultaneous analysis of two independent samples.

9.2.2 GC Temperature Programs and Flow Rates

Table 3 lists the GC operating conditions for the analysis of PCBs as Aroclors for singlecolumn analysis, using wide-bore capillary columns. The GC conditions in these tables are the GC temperature program and flow rates necessary to separate the analytes of interest.

Once established, the same operating conditions must be used for the analysis of samples and standards. Retention times and calibrations will be verified on a daily basis at the beginning of each analytical sequence and retention times will be verified by monitoring subsequent continuing calibration standards.

Note: Once established, the same operating conditions must be used for both calibrations and sample analyses.

Parameter	Settings		
Injector Port Temperature	240°C		
Detector Temperature	325°C		
Temperature Program	100°C for 1 minute 10°C/min to 280°C 20°C /min to 300°C		
Columns 1 and 2 30 m x 0.53 mm ID, 0.5 µm coating			
Injection Volume	2 µL		
Carrier Gas	Helium at 10 mL per minute.		
Make-up Gas 5% Methane in Argon (P5) at 2.5 mL per m			

Table	3	Instrument	Conditions
able	3	instrument	Condition

°C	=	degrees Celsius	ID	=	identification
μL	=	micrograms per liter	mL	=	milliliter
μm	=	micrometers	mm	=	millimeter

9.3 INSTRUMENT CALIBRATION

9.3.1 Initial Calibration

Prepare calibration standards using the procedures in Section 7.5. PCBs will be determined and quantitated as Aroclors using an external standard calibration.

Note: Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.

To establish the calibration factor, estimate the linear range starting at the PQL, which is the lowest concentration that can be accurately quantitated using the established GC analysis conditions. The upper dynamic range of the calibration is dependent on the detector and

operating conditions. Upper calibration standards should demonstrate adequate sensitivity as evaluated using the response factor (RF) for each individual standard. The RF is equal to: RF=Peak Area in the Standard/Total Mass of the Standard Injected (in nanograms).

The initial calibration consists of two parts, described below.

9.3.1.1 Establishment of Linear Dynamic Range

As noted in Section 7.5, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial multi-point calibration is performed using the mixture of Aroclors 1016 and 1260.

9.3.2 Selection of Quantitative Peaks

Sample and standard concentrations will be determined using 5 quantitation peaks for each Aroclor. The peaks must be characteristic of the Aroclor in question. Selected quantitation peaks should be at least 25% of the height of the largest Aroclor peak. The 5 quantitative peaks are selected at the discretion of the analyst, and should demonstrate adequate separation from non-quantitative peaks. When practical, the quantitative peaks should have slopes returning to baseline and not co-elute or shoulder with other peaks. For each Aroclor, the set of quantitation 5 peaks should include at least one peak that is unique to that Aroclor. If the analyst is using the Aroclor 1016/1260 mixture, none of the individual congeners should be found in both of these Aroclors.

Inject 2 μ L of each calibration standard and record the peak area and retention time of each characteristic Aroclor peak to be used for quantitation. Whether using automated or manual integration technique, the peak baseline must be integrated in the same manner as the initial and continuing calibration standards, in order to accurately determine analyte quantities in the sample extract. When five peaks are used for determining sample concentrations, each peak will be assigned a concentration at 1/5th the total concentration in the standard. The

concentration in the sample extract is determined by totaling the concentrations of the five peaks. When field sample peaks do not demonstrate the same characteristics as the standards due to interferences, a peak may be excluded from the quantitation at the discretion of the analyst. The concentration is determined by totaling the concentration of the other four peaks and multiplying the sum by 1.25 in order to normalize the sample concentration. Exclusion of quantitated peaks should only be performed by an experienced analyst after confirmation that the Aroclor has been properly identified, and that no other classes of Aroclors are present in the sample. (See Section 4.0 for description of interferences).

9.3.2.1 Calibration Factors

For a five-point calibration, ten sets of calibration factors will be generated for each standard of the Aroclor 1016/1260 mixture, with each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture. For example, there will be at least 50 separate calibration factors in the multi-point calibration.

9.3.2.2 Establishing the Calibration Function

If a linear calibration model is used, the response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean response or calibration factor, the standard deviation, and the RSD for each Aroclor peak. When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the linear calibration models must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for other Aroclors (such as Aroclor 1254), use the same criteria to evaluate calibration factors from those standards to evaluate linearity. An RSD of less than or equal to 20% is considered an acceptable demonstration of linearity.

Refer to EPA Method 8000B for the specifics of the evaluation of the linearity of the calibration and guidance on performing non-linear calibrations. In general, non-linear calibrations will also consider each characteristic Aroclor peak separately.

9.3.2.3 Qualitative Identification of Other Aroclors

Standards of the other five Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in Section 7.5.1 has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards in Section 7.5.2. These Aroclors must be reinjected if the GC operating conditions are modified, or new columns are installed. If new columns are installed with the same characteristics as the one that is replaced, and no other operating conditions have changed, the analyst may use discretion in determining if the 5 Aroclor standards need to be reinjected. Criteria for the determination include similar retention times and chromatographic patterns nearly identical to those previously established for the qualitative determination of the classes of Aroclor standards.

9.3.2.4 Initial Calibration of Other Aroclor Classes

In situations where other Aroclors of interest are present at a site, the analyst may employ a multi-point initial calibration of the Aroclors of interest (e.g., five standards of Aroclor 1254 if this Aroclor is of concern and linear calibration is employed) and not use the 1016/1260 calibration mixture.

9.4 RETENTION TIME WINDOWS

Absolute retention times are generally used for compound identification. When absolute retention times are used, RTWs are crucial to the identification of target compounds, and should be established by one of the approaches described in EPA Method 8000B.

Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the RTW should be carefully established to minimize the occurrence of both false positive and false negative results. Tight RTWs may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RTWs may result in false positive results that cannot be confirmed upon further analysis. Analysts should reference EPA Method 8000B for the details of

establishing RTWs. Other approaches to compound identification may be employed, provided that the analyst can demonstrate and document that the approaches are appropriate for the intended application. A sum of the area of all peaks (congeners) in any class of Aroclors in not recommended due to the relative inaccuracy of the integration.

When conducting Aroclor analysis, it is important to determine that common singlecomponent pesticides, such as DDT, DDD, and DDE, do not elute at the same retention times as the target congeners. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

9.5 GAS CHROMATOGRAPHIC ANALYSIS OF SAMPLE EXTRACTS

9.5.1 Operating Conditions for Field Samples

The same GC operating conditions used for the initial calibration must be employed for the analysis of all samples and continuing calibration standards.

9.5.2 Continuing Calibration Verification

Verify calibration at least once each 12-hour shift or every 20 samples, by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every 20 samples and at the end of the analysis sequence. For Aroclor analyses, the calibration verification standard will be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition.

9.5.2.1 Continuing Calibration Verification Criteria

The calibration factor for each analyte calculated from the CVS should not exceed a difference of more than ± 20 percent when compared to the mean calibration factor from the initial calibration curve (Refer to Table 3). If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000B for the specifics of calibration verification. % Difference = ((known concentration of standard-standard analytical result/ known concentration) * 100. RF × 100

9.5.2.2 Continuing Calibration Verification Failure

If the calibration does not meet the $\pm 20\%$ limit on the basis of each compound, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within $\pm 20\%$, then a new initial calibration must be prepared. See Section 8.0 for a discussion on the effects of a failing calibration verification standard on sample results.

9.5.3 Qualitative Identification of Aroclors

Qualitative identifications of target analytes are made by examination of the sample chromatograms and comparison of target analytes to known standards injected on the GC under the same analytical conditions.

9.5.4 Quantitative Determination of Aroclor Concentrations

Quantitative results are determined for each identified analyte using the procedures described in Section 9.3 for the external calibration procedure (Method 8000B). If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze.

9.5.5 Sample Bracketing with Continuing Calibration Standards

Each sample analysis employing external standard calibration must be bracketed with an acceptable initial calibration, calibration verification standard(s) after every 10 field samples, or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria in Section 9.3. Multi-level standards are used in the initial calibration to ensure that detector response remains stable for all analytes over the calibration range.

When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent misquantitation and possible false negative results, and reinjection of the sample extracts is required. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis. However, if the standard analyzed after a group of samples exhibits a response for an analyte

that is above the acceptance limit, i.e., >20%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, because the verification standard has demonstrated that the analyte would have been detected if it were present. In contrast, if an analyte above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then reinjection is necessary. The purpose of this reinjection is to ensure that the analyte could be detected, if present, despite the change in the detector response, e.g., to protect against a false negative result.

Sample injections may continue for as long as the CVS and other standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set per EPA Method 8082) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected, after 24 hours of continuous injections, or when qualitative or quantitative QC criteria are exceeded.

9.5.6 Retention Time Stability

Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily RTWs, the system is out of control. Determine the cause of the problem and correct it. Likely causes of retention time shifts are loss of system integrity due to a leaking gas system. Check regulator pressures at the cylinders and flow controls on the GC. If they are the same as the conditions used to initially determine the RTWs, replace the injector septa and/or check for leaks in the system with a helium leak detector.

9.5.7 Analytical Interferences

If compound identification or quantitation is precluded due to interferences (e.g., broad, rounded peaks or ill-defined baselines are present), corrective action is warranted. Cleanup of the extract, column trimming, or replacement of the capillary column or detector may be necessary. The analyst may begin by rerunning the sample on another column to determine if

the problem results from analytical hardware or the sample matrix. Refer to Section 9.1.1 for sample cleanup procedures.

9.6 QUALITATIVE IDENTIFICATION

The identification of PCBs as Aroclors using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the RTWs established through the analysis of standards of the target analytes. See Section 9.4 for information on the establishment of retention time windows. Tentative identification of an Aroclor occurs when peaks from a sample extract fall within the established RTWs for a particular Aroclor.

The results of a single column/single injection analysis may be confirmed, if necessary, on a second, dissimilar, GC column. In order to be used for confirmation, RTWs must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second-column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis. That standard may be the individual Aroclor or the Aroclor 1016/1260 mixture.

When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. This approach should not be attempted for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document:

- The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.
- The absence of major peaks representing any other Aroclor.
- The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.).

Note: This information should either be provided to the data user or maintained by the laboratory.

9.6.1 Confirmation

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily RTW established by injection of a known standard. An experienced analyst must perform the confirmation.

9.7 QUANTITATION OF PCBs AS AROCLORS

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

Once the Aroclor pattern has been identified, compare the response's 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 5 characteristic peaks chosen in Section 9.3 and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. Non-linear calibration may result in different models for each selected peak. A concentration is determined using each of the characteristic peaks and the individual calibration factor calculated for that peak in Section 9.2. Then, these 5 concentrations are totaled to determine the concentration of that Aroclor.

Weathering of PCBs in the environment and changes resulting from chemical or natural weathering processes, may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable.

If results in terms of Aroclors are required, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When

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quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

10.0 GC MAINTENACE

The analytical system must be inspected and maintained on a daily basis to ensure accurate and determinative identification and quantitation of analytical samples.

10.1 METAL INJECTOR BODY

Turn off the oven, cool the detectors and injectors to room temperature, and remove the analytical columns once the oven has cooled. Remove the glass injection port insert. Inspect the injection port and remove any noticeable foreign material.

Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone, and then hexane while catching the rinseate in the beaker.

Deactivating the injector port bodies made of glass injection port liners should be replaced after every 3 days, or as indicated by instrument conditions. Replace the injector liner, reassemble the injector, replace the injector septa, and re-install the columns. Test all fittings with a leak detector to ensure a gas-tight system.

10.2 COLUMN RINSING

Rinse the column with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Fill the column with the appropriate solvent and allow it to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. Afterwards, flush the column with fresh hexane, drain the column, and dry it at room temperature with a stream of ultrapure nitrogen or helium.

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11.0 DATA ANALYSIS AND CALCULATIONS

The determination of sample concentrations is essential to project goals and quality assurance objectives. Whenever possible, spreadsheets with inserted formulas will be utilized to perform routine calculations, including determination of percent solids, sample extract concentrations, and sample concentrations. Sample extract concentrations are determined with Agilent Chemstation/Enviroquant software.

11.1 DETERMINATION OF PERCENT SOLIDS

The determination of the percent solids is performed using a spreadsheet with the following procedures and calculations:

- 1. Zero the 2 place balance.
- 2. Weigh the empty aluminum pan and record the weight.
- 3. Tare the balance with the aluminum pan on the balance.
- 4. Add approximately 10 grams of sample that is representative of the sample. Be sure to remove any rocks or twigs that may be present. Record the weight.
- 5. Place the panned sample in the drying oven, which is set at 104°C, for a minimum of 4 hours or until the sample is dry.
- 6. Remove the dry weight sample and allow to cool to room temperature.
- 7. Record the weight of the dried sample and pan.
- 8. Calculate the percent (%) solids.

Note: % Solids= (dry weight + pan weight)-pan weight)/ wet weight)*100

11.2 DETERMINATION OF SAMPLE CONCENTRATIONS

The concentration in the sample extract is calculated with the data system in Enviroquant. The analyst must ensure that the data system is using the current calibration factors to calculate the concentration of analytes in the extract. The calculation for determining the soil sample concentration is performed on an Excel spreadsheet using the following formula.

Soil concentration= (Concentration of the sample extract $(\mu g/L)/1000 \ \mu g/g)$ X (Volume of the sample extract (5mL of hexane)/dry weight of sample (g)) X dilution factor (1 or more). The result will be in $\mu g/g$, which equates to mg/kg (ppm).

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12.0 METHOD PERFORMANCE

Performance data and related information are provided in EPA SW-846 Solid Waste Methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.1 METHOD DETECTION LIMIT STUDY (MDL)

An MDL study is performed for with the same Aroclor mixture using in the ICAL and spiking solutions, but at a lower concentration. At minimum, the MDL spike should be at or below the PQL. The MDL samples go through the same extraction procedure as field and QC samples. Ten samples are extracted in the same batch along with a method blank. Sample concentrations are quantified and the standard deviation is calculated for all of the MDL samples. The standard deviation is then multiplied by the student T value to determine the MDL.

STANDARD OPERATING PROCEDURE

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13.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

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STANDARD OPERATING PROCEDURE

14.0 WASTE MANAGEMENT

Laboratory waste management practices will be conducted consistently with all applicable federal, state and local rules and regulations. The laboratory will use best practices to protect the air, water, and land, by minimizing and controlling all releases from hoods and bench operations, complying with the all permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult the *Waste Management Plan*, located inside the Bristol Work Plan, which is a separate document. Waste streams will be segregated and stored in categories, such as chlorinated and non-chlorinated solvents, acids and solid waste. Used solvents and acids will be stored in labeled bung top drums. Extracted and unextracted soil and solid reagents, such as sodium sulfate or diatomaceous earth, will be incorporated into the contaminated soil waste stream, which will be disposed of at the appropriate permitted treatment storage disposal facility (TSDF).

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STANDARD OPERATING PROCEDURE

15.0 REFERENCES

- U.S. Environmental Protection Agency (EPA), 2007a. EPA Method 3545A Pressurized Fluid Extraction, Revision 1 and all promulgated updates. EPA Office of Solid Waste. February 2007.
- EPA, 2007b. EPA Method 3550C Ultrasonic Extraction, Revision C and all promulgated updates. EPA Office of Solid Waste. February 2007.
- EPA, 1996 (December). EPA Method 8000B and all promulgated updates. Determinative Chromatographic Separations. EPA Office of Solid Waste. December 1996.
- EPA, 2005 (January). EPA SW846 Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, Fourth addition and all promulgated updates. EPA Office of Solid Waste. January 2005.
- EPA, 2002. Standard Operating Procedure for Polychlorinated Biphenyls (PCBs) Field Testing for Soil and Sediment Samples. The Office of Environmental Measurement and Evaluation. EPA Region New England. 2002.

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February 2010



111 W. 16th Avenue, Third Floor Anchorage, Alaska 99501-5109 907-563-0013 Phone 907-563-6713 Fax

TECHNICAL MEMORANDUM

2010 Field Screening Laboratory and Confirmation Analytical Laboratory Correlation Study for PCB and DRO-RRO Soil Samples

Methodology

Extraction and analytical methods were developed for the field screening laboratory for the quantitation of PCBs and petroleum hydrocarbons (DRO-RRO) in soils at NE Cape St. Lawrence Island. A correlation study was done to compare the field screening laboratory results from soil samples also analyzed by the confirmation analytical laboratory, TestAmerica-Tacoma.

The field screening PCB extraction method was based on an EPA developed rapid extraction method utilizing 1:1 hexane-acetone added to a dehydrated sample inside of a 40 mL VOA vial.

The analytical method is based on EPA SW-846 Method 8082 for the determination of Aroclor concentrations. The DRO-RRO extraction followed the protocols of EPA SW-846 Methods 3545 (Accelerated Solvent Extraction) and EPA Method 3550B (sonication). Method 3550B was used for the correlation samples. Either method may be employed depending on the properties of the soil matrix. Samples with high organic content will be extracted by Method 3550B due to larger sample volumes. The analysis of DRO-RRO samples adhere to Alaska Methods AK102 and AK103.

Sample Selection

PCB samples were obtained from stockpiled soil located on Ft. Richardson Alaska. DRO-RRO samples were obtained from an unrelated project in Nome, Alaska. All samples were sieved through a 2 mm #4 screen and homogenized in a stainless steel bowl. The screened sample was mixed thoroughly for 10 minutes before the soil was placed in jars for analysis.

One jar of PCB contaminated soil and one jar of diesel contaminated soil were submitted to TestAmerica-Tacoma for single analysis as work order #580-19543. Additional jars of homogenized soil were analyzed by Bristol in quadruplicate to establish consistent results and reproducibility. The screening lab and TestAmerica correlation results are included in the Table 1.

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Results

The DRO analyses reported similar concentrations between TestAmerica and the field screening laboratory. TestAmerica reported 17,000 mg/Kg at a 1/10 dilution and the field screening laboratory reported an average of 18,500 for quadruplicate analyses at a 1/5 dilution. The precision between the field screening laboratory and TestAmerica had an RPD of 8.3%. The RRO results had an RPD of 58% with the field screening laboratory reporting a concentration of 2,360 mg/Kg and TestAmerica reporting 1,300 mg/Kg. Both laboratories used the same extraction and analytical methods (3550B and AK102-AK103). The field screening laboratory RSD on 4 samples was 7.9% for DRO and 4.0% for RRO. Surrogate recoveries for the quadruplicate analyses by the field screening laboratory had an average of 85% for DRO (Oterphenyl) and 90% for RRO (n-triacontane d62) with approximately 8% RSD for both DRO and RRO surrogate recoveries.

The PCB analyses had lesser agreement between the field screening laboratory and TestAmerica-Tacoma. The field screening laboratory had an average PCB concentration of Aroclor 1260 at 0.4 mg/Kg and an RSD of 13% with quadruplicate analyses. TestAmerica reported the initial Aroclor 1260 result at 0.2 mg/Kg but with surrogate recoveries below method acceptance limits (38% for Decachlorobiphenyl). The TestAmerica laboratory reported matrix interference as the reason for low surrogate recovery. Bristol requested a re-extraction and re-analysis of the PCB sample and TestAmerica had acceptable surrogate recoveries (74%) but reported an Aroclor 1260 concentration of 0.13 mg/Kg. The low correlation of PCB results between the field screening laboratory and TestAmerica-Tacoma could possibly indicate matrix heterogeneity even though great effort was taken to homogenize the soil during preparation before splitting the sample. This is further evidenced by the low correlation of results from TestAmerica, even with passing surrogate recoveries on the re-extraction and re-analysis, the Aroclor 1260 result was reported at a lower concentration.

The relatively good recoveries of PCBs by the field screening laboratory along with an RSD of 13% for the quadruplicate analyses indicate that the field screening laboratory can consistently perform the method with acceptable precision. All of the method QC such as method blanks, LCS, and LCSD met acceptance criteria for both methods.

Conclusions

The correlation sample results indicate that the field screening laboratory is capable of performing the analyses of POLs and PCBs as well as the confirmation laboratory (TestAmerica-Tacoma). Results from the field screening laboratory will be used to indicate when excavation is sufficient to meet site cleanup levels. As a precaution, field screening laboratory results will use a 20% factor on sample results to indicate when excavation is complete. In other words, a PCB result of 0.8 mg/Kg at the screening lab will be sufficient to stop excavation and collect confirmation samples in order meet the 1 mg/Kg site cleanup level and a DRO result of 7,360 mg/Kg at the field screening laboratory will meet the 9,200 mg/Kg cleanup level for diesel contaminated soils.

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Biogenic interference and silica gel cleanup may be employed when chromatograms indicate a large contribution to the DRO result is from natural organic materials, which are prevalent at the site. Results and chromatograms will be submitted to the USACE project chemist and ADEC for evaluation when biogenic interference is suspected of contributing a large portion of the DRO result. The field screening laboratory chemist is very familiar with biogenic interference and chromatographic interpretation of biogenics.

Sample ID	Dilution	DRO Soil Result	RRO Soil Result	DRO Surrogate Recovery-%	RRO Surrogate Recovery-%
Method Blank	1	ND	ND	78	96
LCS	1	966	1110	84	98
LCSD	1	981	1120	86	91
Corr-1	5	17652	2305	78.8	84.2
Corr-2	5	20558	2497	93.4	98.6
Corr-3	5	18355	2358	88.3	92.9
Corr-4	5	17340	2292	80.6	84.7
Correlation Sa	mple Average				
(mg	/kg)	18500	2360	85.3	90.1
R	elativeStd Dev %	7.9	4.0	8.0	7.7

96

Table 1a Field Screening Laboratory Correlation Study Results

TestAmerica-Tacoma	Results	17000	1300	114
(See Attachment 1)				

	PCB Correlation S	ample Results-Sc	reening Lab	
Sample ID	Dilution	Aroclor 1260 Result (ug/Kg)	TCMX Surrogate Recovery-%	Decachloro- biphenyl Surrogate Recovery-%
Method Blank	1	ND	72	74
LCS	1	966	72	73
LCSD	1	981	70	74
Corr-1	1	440	77	70
Corr-2	1	410	76	70
Corr-3	1	418	83	72
Corr-4*	1	322	63	63
Correlation S	Sample Average (ug/kg)	397	75	69
	RelativeStd Dev %	13	11	6
FestAmerica-Tacoma Re See Attachment 1)	esults	130	96	74

Table 1b Field Screening Laboratory Correlation Study Results

Bristol ENVIRONMENTAL REMEDIATION SERVICES, LLC

ATTACHMENT 1

TestAmerica Analytical Report



ANALYTICAL REPORT

Job Number: 580-19543-1 Job Description: NE Cape

For: Bristol Env. Remediation Services LLC 111 W 16th Ave Suite 301 Anchorage, AK 99501 Attention: Marty Hannah

1. Lone

Approved for release Terri L Torres Project Manager II 6/22/2010 5:34 PM

Terri L Torres Project Manager II terri.torres@testamericainc.com 06/22/2010 Revision: 1

TestAmerica Tacoma is a part of TestAmerica Laboratories, Inc.

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This report shall not be reproduced except in full, without prior express written approval by the laboratory. The results relate only to the item(s) tested and the sample(s) as received by the laboratory.

The results included in this report have been reviewed for compliance with the laboratory QA/QC plan and meet all requirements of NELAC and the DOD QSM V4.1 (4/22/09). All data have been found to be compliant with laboratory protocol, with the exception of any items noted in the case narrative.



Job Narrative 580-19543-1

Receipt

The container label for the following samples did not match the information listed on the Chain-of-Custody (COC): The container cap of sample PCB-CORR-1 lists the time 16:10, while the COC lists the time 15:30. The container cap for sample DRO-CORR-1 lists the time 15:30, while the COC lists the time 16:10.

All other samples were received in good condition within temperature requirements.

GC Semi VOA - Method(s) 8082

Recovery and/or RPD values for PCB-1016 and PCB 1260 in the matrix spike/matrix spike duplicate of sample 580-19543-1 were outside advisory QC limits. Matrix interference is indicated based on acceptable LCS/LCSD recovery and/or RPD.

Recovery of the surrogates from sample 580-19543-1 and the matrix spike of sample 580-19543-1 exceeded quality control limits due to matrix interference.

Sample 580-19543-1 was re-extracted and re-analyzed outside method holding time per client request. Both sets of data are included.

GC Semi VOA - Method(s) AK102 & 103

Recovery values for DRO (nC10-<nC25) and RRO (nC25-nC36) in the matrix spike/matrix spike duplicate of sample 580-19543-1 were outside advisory QC limits. Matrix interference is indicated based on acceptable LCS/LCSD recovery.

METHOD SUMMARY

Client: Bristol Env. Remediation Services LLC

Job Number: 580-19543-1

Description	Lab Location	Method	Preparation Method	
Matrix: Solid				
Polychlorinated Biphenyls (PCBs) by Gas Chromatography	TAL TAC	SW846 8082		
Ultrasonic Extraction	TAL TAC		SW846 3550B	
Alaska - Diesel Range Organics & Residual Range Organics (GC)	TAL TAC	ADEC AK102 &	103	
Ultrasonic Extraction	TAL TAC		SW846 3550B	
Percent Moisture	TAL TAC	EPA Moisture		

Lab References:

TAL TAC = TestAmerica Tacoma

Method References:

ADEC = Alaska Department of Environmental Conservation

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

SAMPLE SUMMARY

Client: Bristol Env. Remediation Services LLC

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
580-19543-1	PCB-CORR-1	Solid	05/24/2010 1530	05/26/2010 0940
580-19543-2	DRO-CORR-1	Solid	05/24/2010 1610	05/26/2010 0940

Client: Bristol Env. Remediation Services LLC

Client Sample ID:	PCB-CORR-1				
Lab Sample ID: Client Matrix:	580-19543-1 Solid	% Moisture: 13.3	3		Sampled: 05/24/2010 1530 Received: 05/26/2010 0940
	8082 Polyc	hlorinated Biphenyls (PCBs)	by Gas Chro	matography	- 1990-
Method:	8082	Analysis Batch: 580-64551		Instrument ID:	TAC034
Preparation:	3550B	Prep Batch: 580-64511		Initial Weight/Volume:	10.3904 g
Dilution:	1.0			Final Weight/Volume:	10 mL
Date Analyzed:	05/27/2010 1808			Injection Volume:	1 uL
Date Prepared:	05/27/2010 1156			Result Type:	PRIMARY
Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	DL	LOQ
PCB-1016	nannan a san a san an san ann an san san	0.008879	UJ	0.0036	0.011
PCB-1221		0.01110	U	0.0089	0.011
PCB-1232		0.01110	U	0.0078	0.011
PCB-1242		0.006659	U	0.0023	0.011
PCB-1248		0.003330	U	0.0014	0.011
PCB-1254		0.006659	U	0.0023	0.011
PCB-1260		0.20	J	0.0033	0.011
Surrogate		%Rec	Qualifier	Accepta	nce Limits
Tetrachloro-m-xyler		38	J	45 - 155	na (delenana y manana antio d'an ana ana anna anna an an an an an an a
DCB Decachlorobip		41	J	60 - 125	

Client: Bristol Env. Remediation Services LLC

Client Sample ID:	PCB-CORR-1				
Lab Sample ID:	580-19543-1			Date	Sampled: 05/24/2010 1530
Client Matrix:	Solid	% Moisture: 13.3		Date	Received: 05/26/2010 0940
528	8082 Poly	chlorinated Biphenyls (PCBs) b	y Gas Chromato	graphy	
Method:	8082	Analysis Batch: 580-66015	Instr	ument ID:	TAC042
Preparation:	3550B	Prep Batch: 580-65896	Initia	Weight/Volume:	10.2641 g
Dilution:	1.0		Fina	Weight/Volume:	10 mL
Date Analyzed:	06/21/2010 1652	Run Type: RE		tion Volume:	1 uL
Date Prepared:	06/18/2010 1529			ult Type:	SECONDARY
Analyte	DryWt Corrected: `	Y Result (mg/Kg)	Qualifier	DL	LOQ
PCB-1016		0.0090	UH	0.0036	0.011
PCB-1221		0.011	UH	0.0090	0.011
PCB-1232		0.011	UH	0.0079	0.011
PCB-1242		0.0067	UH	0.0024	0.011
PCB-1248		0.0034	UH	0.0015	0.011
PCB-1254		0.0067	UH	0.0024	0.011
PCB-1260		0.13	н	0.0034	0.011
Surrogate		%Rec	Qualifier	Acceptar	nce Limits
Tetrachloro-m-xyler	le	94	i - Janeira - Calder Back II. 1996, ar la far taga a thigh sa sa an an an an an	45 - 155	nin disensi susinin ya palentari wa misi disebuti sa chima kunini dashi nchi su wa shabari nce sang dise
DCB Decachlorobip	bhenyl	74		60 - 125	
	,			00 120	

Client: Bristol Env. Remediation Services LLC

Client Sample ID:	DRO-CORR-1				
Lab Sample ID:	580-19543-2			Date	Sampled: 05/24/2010 1610
Client Matrix:	Solid	% Moisture: 14.	2	Date	Received: 05/26/2010 0940
	AK102 & 103 Alas	ka - Diesel Range Organics	& Residual Range	e Organics (GC)	- 9880
Method:	AK102 & 103	Analysis Batch: 580-64595	5 Ins	strument ID:	TAC019
Preparation:	3550B	Prep Batch: 580-64501	Lal	b File ID:	GR03125.D
Dilution:	1.0		Init	tial Weight/Volume:	10.4425 g
Date Analyzed:	05/28/2010 1321		Fin	al Weight/Volume:	10 mL
Date Prepared:	05/27/2010 1036		Inje	ection Volume:	1 uL
Analyte	DryWt Corrected: Y	Result (mg/Kg)	Qualifier	DL	LOQ
RRO (nC25-nC36)	nd yn arwedd yn ar yn	1300		12	56
Surrogate		%Rec	Qualifier	Accepta	nce Limits
o-Terphenyl		114		60 - 120	Non des als des many and and and an
n-Triacontane-d62		96		60 - 120	

Client: Bristol Env. Remediation Services LLC

Client Sample ID:	DRO-CORR-1						
Lab Sample ID:	580-19543-2				Date	Sampled: 05/24/2010 1	1610
Client Matrix:	Solid	% Moisture:	14.2		Date	Received: 05/26/2010 0)940
	AK102 & 103 Alas	ka - Diesel Range Orga	nics & Resid	ual Range	Organics (GC)		
Method:	AK102 & 103	Analysis Batch: 580-64	1595	Instr	ument ID:	TAC019	
Preparation:	3550B	Prep Batch: 580-6450	1	Lab	File ID:	GR03143.D	
Dilution:	10			Initia	I Weight/Volume:	10.4425 g	
Date Analyzed:	05/28/2010 1715			Fina	I Weight/Volume:	10 mL	
Date Prepared:	05/27/2010 1036			Injec	tion Volume:	1 uL	
Analyte	DryWt Corrected: Y	Result (mg/Kg) Q	ualifier	DL	LOQ	
DRO (nC10- <nc25< td=""><td></td><td>17000</td><td></td><td>enderstrade alle the set of the flatter of the game</td><td>26</td><td>220</td><td>NO DIVERSI MINU</td></nc25<>		17000		enderstrade alle the set of the flatter of the game	26	220	NO DIVERSI MINU

Client: Bristol Env. Remediation Services LLC

			Gene	eral Chemi	istry			
Client Sample ID:	PCB-CORR-1							
Lab Sample ID: Client Matrix:	580-19543-1 Solid							ed: 05/24/2010 1530 ed: 05/26/2010 0940
Analyte		Result	Qual	Units	LOQ	LOQ	Dil	Method
Percent Solids	Analysis Batch: 580-6	87 64400	Date Analyzed	% : 05/26/201	0.10 10 1158	0.10	1.0	Moisture DryWt Corrected: N
Percent Moisture	Analysis Batch: 580-	13 64400	Date Analyzed	% 05/26/201	0.10 10 1158	0.10	1.0	Moisture DryWt Corrected: N

			Gen	eral Chemi	stry			
Client Sample ID:	DRO-CORR-1							
Lab Sample ID:	580-19543-2						Date Sample	ed: 05/24/2010 1610
Client Matrix:	Solid		•				Date Receiv	ed: 05/26/2010 0940
Analyte		Result	Qual	Units	LOQ	LOQ	Dil	Method
Percent Solids	Carl Property and a submitter second consideration of the submitter of the	86	ala anticipa de la tipo de la contra de la 1979 e dante esta como nomen de la compositiva de la contra de la c	%	0.10	0.10	1.0	Moisture
	Analysis Batch: 580-	64400	Date Analyzed	05/26/201	0 1158			DryWt Corrected: N
Percent Moisture		14		%	0.10	0.10	1.0	Moisture
	Analysis Batch: 580-	64400	Date Analyzed	05/26/201	0 1158			DryWt Corrected: N

Job Number: 580-19543-1

Method: 8082 Preparation: 3550B

Initial Weight/Volume:

10 g

Client: Bristol Env. Remediation Services LLC

Method Blank - Batch: 580-64511

	MB 580-64511/1-A	Analysis Batch: 580-64551		Instrument ID: TAC034
	Solid	Prep Batch: 580-64511		Lab File ID: PCB27839.D
Dilution:	1.0	Units: mg/Kg		Initial Weight/Volume: 10 g
Date Analyzed:	05/27/2010 1722			Final Weight/Volume: 10 mL
Date Prepared:	05/27/2010 1156			Injection Volume: 1 uL
				Column ID: PRIMARY
Analyte		Result	Qual	DL LOQ
PCB-1016		0.008000	U	0.0032 0.010
PCB-1221		0.01000	U	0.0080 0.010
PCB-1232		0.01000	U	0.0070 0.010
PCB-1242		0.006000	U	0.0021 0.010
PCB-1248		0.003000	U	0.0013 0.010
PCB-1254		0.006000	U	0.0021 0.010
PCB-1260		0.008000	U	0.0030 0.010
Surrogate	Y ₁₆ ma	% Rec		Acceptance Limits
Tetrachloro-m-xyle	ene	79		45 - 155
DCB Decachlorobi	iphenyl	85		60 - 125
Lab Control Sa	mple/			Method: 8082
Lab Control Sa	mple Duplicate Recovery	Report - Batch: 580-64511		Preparation: 3550B
LCS Lab Sample I	ID: LCS 580-64511/2-A	Analvsis Batch: 580-64551		Instrument ID: TAC034
Contract of the second				Lab File ID: PCB27840.D
Client Matrix:	Solid	Prep Batch: 580-64511		Lau File ID. FUD2/040.D

10	9 105 LCS % F		30 3 SD % Rec	20 Accepta	nce Limits	
10	9 105	60 - 1	30 3	20		
10		00 44	0 0	00		
99	95	40 - 14	40 4	20		
. LC	LCS	SD Limit	RPD	RPD Limit	LCS Qual LCSD Qual	
	% Rec.					
				Column ID:	PRIMARY	
27/2010 1156				Injection Volume:	1 uL	
27/2010 1753	•			Final Weight/Volume:		
l	Jnits: mg/K	g		Initial Weight/Volume: 10 g		
d I	Prep Batch:	580-64511		Lab File ID: PCB	27841.D	
CSD 580-64511/3-A	Analysis Batc	h: 580-64551		Instrument ID: T	AC034	
				Column ID:	PRIMARY	
27/2010 1156				Injection Volume:	1 uL	
27/2010 1737				Final Weight/Volume:	10 mL	
	7/2010 1737 7/2010 1156 SD 580-64511/3-A	7/2010 1737 7/2010 1156 SD 580-64511/3-A Analysis Batch d Prep Batch: Units: mg/K	7/2010 1156 SD 580-64511/3-A Analysis Batch: 580-64551 d Prep Batch: 580-64511 Units: mg/Kg	7/2010 1737 7/2010 1156 SD 580-64511/3-A Analysis Batch: 580-64551 d Prep Batch: 580-64511 Units: mg/Kg	7/2010 1737 Final Weight/Volume: 7/2010 1156 Injection Volume: Column ID: Column ID: SD 580-64511/3-A Analysis Batch: 580-64551 Instrument ID: The prep Batch: 580-64511 Lab File ID: PCB2 Units: mg/Kg Initial Weight/Volume:	

Units: mg/Kg

Dilution:

1.0

Client: Bristol Env. Remediation Services LLC

Job Number: 580-19543-1

Matrix Spike/

Matrix Spike Duplicate Recovery Report - Batch: 580-64511

Method: 8082 Preparation: 3550B

60 - 125

Tetrachloro-m-xylene			40	J 57		4	5 - 155	
Surrogate			MS % Rec	MSD 9	% Rec	Acce	ptance Limits	
PCB-1260		37	24	60 - 130	22	20	J	J
PCB-1016	ne o par ann ann an an ann an Anna an Anna an Anna ann an Anna	8	14	40 - 140	7	20	J	J
Analyte		<u>%</u> МS	<u>Rec.</u> MSD	Limit	RPD	RPD Limit	MS Qual	MSD Qual
Date Prepared:	05/27/2010 1156					ijection Volume: olumn ID:	1 uL PRIMARY	,
Date Analyzed:	05/27/2010 1839					inal Weight/Volur		L
Dilution:	1.0				In	itial Weight/Volu	me: 10.207	'3 g
Client Matrix:	Solid	Prep	Batch: 580-6	64511	La	ab File ID: P	CB27844.D	
MSD Lab Sample ID:	580-19543-1	Analy	sis Batch: 5	80-64551	In	strument ID: T	AC034	
						olumn ID:	PRIMARY	
Date Prepared:	05/27/2010 1156					jection Volume:	1 ul	
Date Analyzed:	05/27/2010 1823					inal Weight/Volu		•
Dilution:	1.0					itial Weight/Volu		331 g
MS Lab Sample ID: Client Matrix:	580-19543-1 Solid		sis Batch: 5 Batch: 580-6				TAC034 PCB27843.D	

61

J

42

DCB Decachlorobiphenyl

Job Number: 580-19543-1

Client: Bristol Env. Remediation Services LLC

Method Blank - Batch: 580-65896

Method Blank - Ba	ICN: 580-65896					Preparation: 3550	B	
Client Matrix: So Dilution: 1.0 Date Analyzed: 06	3 580-65896/1-A Jid) /21/2010 1540 /18/2010 1517	•	s Batch: 580- atch: 580-658 mg/Kg		L Ir F	ab File ID: CC nitial Weight/Volum inal Weight/Volume njection Volume:	0	r.
Analyte			Result		Qual	DL	LOC	2
PCB-1016	nan fan fan i gener af en an en de anger er anne an er an yna y gener af gener er gener er an er en er en er an	an ann Chairte Alan a shearain a she	0.0080		U	0.0032	0.01	0
PCB-1221			0.010		U	0.0080	0.01	0
PCB-1232			0.010		U	0.0070	0.01	0
PCB-1242			0.0060		U	0.0021	0.01	0
PCB-1248			0.0030		U	0.0013	0.01	
PCB-1254			0.0060		U	0.0021	0.01	
PCB-1260			0.0080		U	0.0030	0.01	
Surrogate			% Rec			Acceptance Limits	i.	
Tetrachloro-m-xylene	2		100			45 - 155		
DCB Decachlorobiph			94			60 - 125		
		Arrah	-is Databa 50	0.00045			0040	
LCS Lab Sample ID:			sis Batch: 58				C042	
Client Matrix:	Solid		Batch: 580-6	5896			150049.D	
Dilution:	1.0	Units:	mg/Kg			tial Weight/Volume	-	
Date Analyzed:	06/21/2010 1554				Fir	nal Weight/Volume:	10 r	nL
Date Prepared:	06/18/2010 1517					ection Volume: Jumn ID:	1 uL PRIMARY	
LCSD Lab Sample I	D: LCSD 580-65896/7-A	Analy	sis Batch: 58	0-66015	Ins	strument ID: T	AC042	
Client Matrix:	Solid	Prep	Batch: 580-6	5896	La	b File ID: CC1	50054.D	
Dilution:	1.0		mg/Kg		Ini	tial Weight/Volume	: 10 g	
Date Analyzed:	06/21/2010 1706		0.0			al Weight/Volume:	-	
Date Prepared:	06/18/2010 1529					ection Volume:	1 uL	-
Dute i repared.						lumn ID:	PRIMARY	
			% Rec.					
Analyte		LCS	LCSD	Limit	RPD	RPD Limit	LCS Qual	LCSD Qual
PCB-1016		75	75	40 - 140	1	20		a por tita a tanàna dia dia mampina dia mandritra dia mandritra dia mandritra dia mandritra dia mandritra dia m
PCB-1260		84	81	60 - 130	4	20		
Surrogate			CS % Rec		% Rec	Accepta	nce Limits	n Name (Presses SPRC) a star Perio e albite (Sprt Perio)
Tetrachloro-m-xylene			99	102			- 155	

Method: 8082

TestAmerica Seattle

DCB Decachlorobiphenyl

87

60 - 125

93

Client: Bristol Env. Remediation Services LLC

Job Number: 580-19543-1

Lab Sample ID:MB 580-64501/1-AClient Matrix:SolidDilution:1.0Date Analyzed:05/28/2010 1204Date Prepared:05/27/2010 1036	Analysis Batch: 580-64595 Prep Batch: 580-64501 Units: mg/Kg		Instrument ID: TAC019 Lab File ID: GR03119.D Initial Weight/Volume: 10 g Final Weight/Volume: 10 mL Injection Volume: 1 uL		
Analyte	Result	Qual	DL	LOQ	
DRO (nC10- <nc25)< td=""><td>6.2</td><td>U</td><td>2.3</td><td>20</td></nc25)<>	6.2	U	2.3	20	
RRO (nC25-nC36)	32	U	11	50	
Surrogate	% Rec		Acceptance Limits		
o-Terphenyl	84		60 - 120		
n-Triacontane-d62	77		60 - 120		

Lab Control Sample/

Lab Control Sample Duplicate Recovery Report - Batch: 580-64501

Method: AK102 & 103 Preparation: 3550B

Method: AK102 & 103 Preparation: 3550B

Client Matrix: S Dilution: 1 Date Analyzed: 0	CS 580-64501/2-A Solid 1.0 15/28/2010 1230 15/27/2010 1036	Analysis Batch: 580-64595 Prep Batch: 580-64501 Units: mg/Kg	Instrument ID: TAC019 Lab File ID: GR03121 Initial Weight/Volume: Final Weight/Volume: Injection Volume:	1.D 10 g 10 mL 1 uL
Client Matrix:SDilution:1Date Analyzed:0	LCSD 580-64501/3-A Solid .0 95/28/2010 1256 95/27/2010 1036	Analysis Batch: 580-64595 Prep Batch: 580-64501 Units: mg/Kg	Final Weight/Volume:	

	9	6 Rec.					
Analyte	LCS	LCSD	Limit	RPD	RPD Limit	LCS Qual	LCSD Qual
DRO (nC10- <nc25)< td=""><td>88</td><td>88</td><td>75 - 125</td><td>1</td><td>20</td><td></td><td></td></nc25)<>	88	88	75 - 125	1	20		
RRO (nC25-nC36)	91	92	60 - 120	2	20		
Surrogate	LCS % Rec		LCSD % Rec		Acceptance Limits		
o-Terphenyl	8	8	85		6	0 - 120	
n-Triacontane-d62	8	5	81		6	0 - 120	

Client: Bristol Env. Remediation Services LLC

Matrix Spike/	Method: AK102 & 103				
Matrix Spike Duplicate Recovery Report - Batch: 580-64501	Preparation: 3550B				

580-19543-2	Analysis Batch: 580-64595	Instrument ID: TAC019
Solid	Prep Batch: 580-64501	Lab File ID: GR03127.D
1.0		Initial Weight/Volume: 10.3863 g
05/28/2010 1347		Final Weight/Volume: 10 mL
05/27/2010 1036		Injection Volume: 1 uL
580-19543-2	Analysis Batch: 580-64595	Instrument ID: TAC019
Solid	Prep Batch: 580-64501	Lab File ID: GR03129.D
1.0		Initial Weight/Volume: 10.3461 g
05/28/2010 1413		Final Weight/Volume: 10 mL
05/27/2010 1036		Injection Volume: 1 uL
	Solid 1.0 05/28/2010 1347 05/27/2010 1036 580-19543-2 Solid 1.0 05/28/2010 1413	Solid Prep Batch: 580-64501 1.0 05/28/2010 1347 05/27/2010 1036

	<u>%</u>						
Analyte	MS	MSD	Limit	RPD	RPD Limit	MS Qual	MSD Qual
DRO (nC10- <nc25)< td=""><td>2570</td><td>2560</td><td>72 - 128</td><td>0</td><td>20</td><td>J</td><td>J</td></nc25)<>	2570	2560	72 - 128	0	20	J	J
RRO (nC25-nC36)	295	298	53 - 116	1	21	J	J
Surrogate		MS % Rec	MSD 9	% Rec	Acce	eptance Limits	
o-Terphenyl		105	112		6	0 - 120	
n-Triacontane-d62		96	92		6	0 - 120	

DATA REPORTING QUALIFIERS

Client: Bristol Env. Remediation Services LLC

Lab Section	Qualifier	Description	
GC Semi VOA			
	J	Estimated: The quantitation is an estimation due to discrepancies in meeting certain analyte-specific quality control criteria.	
	н	Sample was prepped or analyzed beyond the specified holding time	
	U	Undetected at the Limit of Detection.	

Seame/Ta

5755 8th Street East

Tacoma, WA 98424

Chain of Custody Record

16	es	t¥	t	n	e	ic	ca
出版的		in status	11 March	副常語			
THE	LEAD	ER IN	ENV	IRON	MENT		ESTING

hone 253.922.2310 fax 253.922.5047 TestAmerica Laboratories, Inc.																								
Client Contact	Project Ma										5/25/2	2010				COC 1	No:							
Bristol Environmental							Lab Contact: Terri Torres							Carrier: Fed Ex							of	_ COCs		
111. W. 16th St		Analysis T																	Job N					
Anchorage, AK 99501		(C) or Wor	W		· 推進															195	43			
907-563-0013	1	T if different f			濡																10			
(xxx) xxx-xxxx FAX		2			The second secon														SDG	No.				
Project Name: NE Cape		1			聖儀																			
Site:	x			e																				
P O # 410026		1	day			amp	1082	loist															_	
Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.	Filtered S	PCBs by 8082	Percent Moistu													Sample Sj	pecific No	otes:	
PCB-CORR-1	5/24/2010	1530	COMP	s	1	2	x	x																
DRO-CORR-1	5/24/2010	1610	COMP	s	1		х	x																
						Ш																		
Раде																								
ō																								
17																								
O th		30) 																						
20			1																					
																				×.				
																							_	
Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other																								
Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaO Possible Hazard Identification Non-Hazard Flammable Skin Irritant	Poison B Unknown								Dispo turn 1						ssessed if samples are retained					ed longe ve For		o nth) Months		
		2			том	ART		Nel		0.01	on		<u> </u>	nap0.	sai Dj	Lab			AUGIII			monuns		-
Special Instructions/QC Requirements & Comments: PLEASE PROVIDE PERCENT SOLIDS REPORTING TO MARTY																								
Relinquished by	Company:	Date/Time: 10:00			KVION							Company:						Date/I	5/26/	1D	094	10		
Relinquished by:	Company:	Date/Time:			Received by:							Company:						Date/1	ìmé:					
Relinquished by:	Company:		Date/Tir	ne:	.]	. Received by:							Company:						Date/1	Date/Time:				

Cool	ler ID No. <u>SM fled just</u> TAL Work Ord	ler <u>19543</u>
	COOLER RECEIPT FORM	
Proj	ect NE Cape	
Coo	ler received on <u>5/26</u> and opened on <u>5/26</u> by <u>(F)</u>	
	Kherle	24
	(signature) perature upon receipt: Cooler <u>2 4</u> oC.	
Tem	perature upon receipt: Cooler <u>29</u> oC. Temp. Blank <u>0.6</u> oC.	
1.	Were custody seals on outside of cooler and intact?	YESNO
	 a. If yes, how many and where: <u>Hronk</u> b. Were signature and date correct? 	
2.	Were custody papers taped to lid inside cooler?	YES NO
3.	Were custody papers properly filled out(ink, signed, etc)?	YESNO
4.	Did you sign custody papers in the appropriate place?	YESNO
5.	Did you attach shipper's packing slip to this form?	YES NO
6.	What kind of packing material was used? <u>Jee facks</u>	-
7.	Was sufficient ice used?	YES NO
8.	Were all bottles sealed in separate plastic bags?	YES NO
9.	Did all bottles arrive in good condition (unbroken)?	YES NO
10.	Were all bottle labels complete (no., date, signed, pres, etc)?	YESNO
11.	No info Ottlabels, Only JDison lids Did all bottle labels and tags agree with custody papers?	YES NO NA
12.	Were correct bottles used for the test indicated?	YES NO
13.	If present, were voa vials checked for absence of airbubbles and noted if found?	YES NO
14.	Adequate volume of voa vials received per sample?	YES NO /
15.	Was sufficient amount of sample sent in each bottle?	YES NO
16.	Were correct preservatives used?	YES NO
17.	Were extra labels added to pre-tared containers?	-YES-NO-NA
18.	Corrective action taken, if necessary: a. Name of person contacted: b. Date:	

From: Origin ID: CYMA (907) 563-0013 Ship Date: 25MAY10 Fed Ex ActWgt: 6.0 LB CAD: 5507712/INET3010 Dims: 10 X 7 X 7 IN Tammy Miller Bristol Enviro Remediation 111 W 16th Ave Suite 301 Delivery Address Bar Code Anchorage, AK 99501 007220224 SHIP TO: (253) 922-2310 **BILL SENDER** Ref# 410026 **Terri Torres** Invoice # PO# TestAmerica Dept # 5755 8TH ST E EAST FIFE, WA 98424 WED - 26 MAY A2 TRK# 0201 7986 9688 0099 STANDARD OVERNIGHT 98424 WA-US WV TCMA SEA -7. 2 After printing this label: page to print your label to your laser or inkiet printer. 1. U~~ nt' button an this 2. Fi 3. Pi el can be read and scanned. War ing purposes is fraudulent and could resul stody Sea Use d available on fedex.com.FedEx will not be wery, misdelivery, or misinformation respo. you'de uss and file a timely claim. Limitations for Guide any loss, including intrinsic valueof the costs, remistor damage whether direct, incidental, consequential Recovery cannot ed actual documented loss.Maximum for items of ex instruments a er items listed in our ServiceGuide. Written claims mus ċ CI. stady Seal 9 https://www.fedex.com/shipping/html/en//PrintlFrame.html 5/2.5/2.010

Login Sample Receipt Check List

Client: Bristol Env. Remediation Services LLC

Job Number: 580-19543-1

Login Number: 19543 Creator: Presley, Kim		List Source: TestAmerica Tacoma
List Number: 1		
Question	T / F/ NA	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
There are no discrepancies between the sample IDs on the containers and the COC.	False	Times on labels are switched w/ times on COC.
Samples are received within Holding Time.	True	
Sample containers have legible labels.	False	Labels are not written on, Info written on caps.
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	N/A	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Is the Field Sampler's name present on COC?	False	no
Sample Preservation Verified	N/A	

Reviewer: ADEC-Curtis Dunkin Re: ADEC post review conf. draft final comments on 2010 NECape HTRW WP and SAP Submitted 7/1/2010

F

#	Reference	ADEC Comment	Bristol Response	USACE Response
1.	p.14, Sec3.2.5 HTRW WP	States Bristol will develop/implement SAP to MNA parameters and to collect surface water samples. Should this state "Bristol has developed and is included in the SAP" or does this refer to something else other than what is listed in the SAP for Site 8?	Statement will be corrected to state that 'Bristol has developed and will implement a sampling and analysis plan to monitor(see SAP Section 3.4)	
2.	р. 15, Sec3.2.6 HTRW WP	During the review conf. on June 23, 2010, ADEC requested samples to be taken within the source area of Site 9. Please address.	Currently Bristol is scoped to collect surface water samples at 4 locations before, during, and after the landfill cap is complete. Three locations are in the stream flowing through the landfill and to the Suqi. The 4 th location is on the Suqi River just above the confluence with the Site 9 stream.	Additional <u>soil</u> sampling from within Site 9 is not planned. If hazardous items such as batteries are identified during capping, they will be removed. The objective is to mitigate potential exposure, not dig
3.	p. 19 Site 28 General HTRW WP/ SAP	What are the criteria for determining/deciding whether manhole and/or culvert will be cleaned, removed, and/or plugged? If removed (resulting in soil and/or sediment disturbance), confirmation surface water, soil and/or sediment samples should be taken to characterize potential residual contamination. Does any of the mentioned 12" corrugated metal pipe remain, and if so, how will that be addressed in the event of manhole removal? ADEC expressed during the June 23, 2010 review conf. whether it was appropriate to work on the manhole/culvert portion of site 28 until the remainder/entirety of site 28 was addressed, given potential release of contamination. Please address.	 The removal of the manhole and plugging of the culvert will remove these potential point sources to the Site 28 Drainage Basin. In the meeting we discussed this area is already heavily contaminated and may still be impacted from surface runoff and subsurface seeps from the MOC until the contaminated soil at the MOC and Site 28 Drainage Basin are remediated in the future. Bristol is planning to completely remove the manhole, sample sediment from within the manhole (for disposal purposes), and dispose of the sediment and manhole offsite. Bristol plans to inspect the culvert and dig down to determine the exact size of the culvert. Bristol will plug it with bentonite and cap it with a manufactured cap that fits the culvert size. Bristol does not know if any of the 12" CMP remains. Bristol will discuss this with the QAR at the time we remove the manhole and contact the ADEC with any additional information. 	into the landfill area Additional confirmation sampling is not necessary because there is known residual contamination at the site which has already been characterized. Soil/sediment removal at this site is scheduled in the future.

4.	p.24 Sec4.1.4.3	"Site 600" appears to be a typo, change to Site 6 (same typo p.15 of SAP). How will oversized material be sampled to obtain representative samples? How will oversized rock samples be crushed? How will stained/contaminated oversized frozen material/clumps be separated from non-stained oversized rock? What will be the maximum volume of individual stockpiles of oversized material derived from PCB contaminated areas to minimize sample dilution?	Site 600 will be corrected to read Site 6 in the SPA and WP.Text will be added to state that at least one rock sample will be obtained from each 10 cubic yards of stockpiled oversized material for field screening purposes; rock samples will be obtained from various depths in the pile. At least 2 rock samples will be collected from stockpiles of 50 cubic yards or less, with at least one additional sample collected from each additional 50 cubic yards of the stockpile or portion thereof over the initial 50 cubic yards. The rock samples will be sent to Dowl-Alaska Testlab to be crushed, and then shipped to TestAmerica-Tacoma for PCB analysis.The field crew assigned to the screen plant in cooperation with the QAR will determine when truck loads of PCB contaminated soil should be placed directly into bulk bags and not screened due to heavily stained rocks, and/or when frozen material is incorporated with the PCB contaminated soil.
5.	Site 8 General HTRW WP/ SAP	How are grids being selected? Plans should include figure showing prior sample locations and results, and proposed 2010 grids. During the first year of sampling, discreet samples would provide a more statistically valid baseline; composite samples could be conducted in subsequent years. How will extent of impacted area be delineated/surveyed?	Grids will be selected by a random number generator, using an Excel program. The 2 sediment samples (08SD103 and 08SD102) and the surface water sample (08SW101) and results will be included as well as the proposed grid and decision units on the figure. Bristol believes that the composite samples will be more valid based on results from multi-incremental soil sample which uses composite samples. The impacted area will be determined through field screening, and visual and olfactory senses.
6.	p.26 SAP	States table 3.6cont. – appears to be 3.7cont.	Comment acknowledged. Table numbering and formatting will be corrected when these documents go to production of the hardcopies

APPENDIX D

Documentation Forms

Groundwater Low Flow Purging Form Groundwater Sampling Information Form Photograph Log Sample Label Sample Receipt Checklist Sample Record Log TestAmerica Sample Receipt TestAmerica Chain-of Custody Record Form ENVIRONMENTAL REMEDIATION SERVICES, LLC

Bristol

GROUNDWATER LOW-FLOW PURGING FORM

Job Name			Well	No.:						
Job Numbe	er		Well	Туре:	Monitor		🗌 Exti	raction	Other	
Company			Well	Material	PVC		St. Steel Other			
			Date					Ti	me:	
Purged by					_					
						Signature)				
				WEI	L PURGIN	G				
PURGE VOLUN	1E					ETHOD				
Casing Diameter (D	in inches):				🗌 Pump – T	ype:				
☐ 4-] 2-inch inch	6-inch	□ Ot	her		Submersi	ble 🗌 C	entrifugal	🗌 Bla	Idder Per	istaltic.
otal Depth of Casin		Other – T	ype:							
Vater Level Depth (PUMP INT	AKE SE	TTING						
					☐ Near Bottom	□ Ne	ear Top	Other		
					Depth in feet	(BTOC):		Scr	een Interval i	n Feet (BTOC)
PURGE 1	IME				PURGE RA	ΓE		ACTU	AL PURG	E VOLUME
Start		Stop	Elap	sed	Initial	gpm	Final	-	_ gpm	gallons
IELD PARAME	TER MEAS	UREMEN	т							
Minutes Since Pumping Began	Water Depth below MP	Pump Dial	Purge Rate (ml/min)	T □ °C □ °F	Specific Cond. (µS/cm)	рН	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Cumulative Volume Purged

GROUNDWATER LOW-FLOW PURGING FORM (continued)

FIELD PARAMETER MEASUREMENT (Continued)

Minutes Since Pumping Began	Water Depth below MP	Pump Dial	Purge Rate (ml/min)	T □ °C □ °F	Specific Cond. (µS/cm)	рН	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Cumulative Volume Purged
								+		

O: Jobs/410026 NE Cape HTRW/10 Mgmt/Planning Documents/SAP/Appendix D Documentation Forms/GROUNDWATER LOW FLOW PURGING FORM.doc

	IRONMENTAL IEDIATION SER	VICES, LLC							
Job Name Job Number		Date			Time:				
Recorded by		Duic							
		WELL	INFORMATIO	N					
ell Number			Well Location						
	nches):		Total Depth of Ca	using (TD in feet BTC	DC):				
asing Diameter (D in ii									
asing Diameter (D in ii] 2-inch	☐ 6-inch ☐ O	ther	Water Level Dep	h (WL in feet BTOC)):				
] 2-inch 🔲 4-inch	☐ 6-inch ☐ O		Water Level Dep	h (WL in feet BTOC)	<u>.</u>				
2-inch 4-inch AMPLING METH Bailer – Type:	□ 6-inch □ O OD □ Centrifugal □	WEL	L SAMPLING						
] 2-inch 4-inch AMPLING METH] Bailer – Type:	□ 6-inch □ O OD □ Centrifugal □	WEL	L SAMPLING	Гуре					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0 OD □ Centrifugal □	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch	□ 6-inch □ 0	WEL] Bladder	L SAMPLING	Type] Other – Type:					
2-inch 4-inch AMPLING METH Bailer – Type: Submersible Submersible Sample No. Sample No.	□ 6-inch □ 0	WEL] Bladder	L SAMPLING	Type] Other – Type:	Lab				

O: Jobs \410026 NE Cape HTRW \10 Mgmt \Planning Documents \SAP \Appendix D Documentation Forms \GROUNDWATER SAMPLING INFO to accompany Low Flow Purging Form.doc

PHOTOGRAPH LOG

DATE	TIME	LOCATION	DESCRIPTION OF PHOTOGRAPH	VIEW DIRECTION	PHOTOGRAPHER/COMMENTS

SAMPLE LABEL

2010 NE Cape HTRW Remedial Actions Contract No. W911-KB-10-C-0002

Analysis:	Preservation:
Date:	Time:
Collector:	
Sample No.:	

TestAmerica Sample Receipt Checklist

Received by:	Unpacked by'	Logged-in by:	Work Or	rder No			
(section 4)	(section B)		Client:				
Date:	Date:	Date	Project:				
Time:	Initials:	Initials:					
A Custody Seals;	Cooler Temperature (IR):	C plastic	glass NA <i>(oil/air samples, E</i>	Temparatur SI client)	e out of ran	ge No fee fee Me Wiin 4 Other.	fted
A Custody Seals:			В	Sample Status:			
Signature: Y N Dated:	Deviet			(If N circled, see No			
None	Received by:			(III V CIICIEU, SEE IV	50)		
Container Type:		A Courier	General: Intact?		V	N.	
#Cooler(s)	Sc	-	indot:		Y	N	
#Box(s)	U		# Containers Ma	atch COC?	У	N	none given
#Other:	Fe		IDs Match COC	?	У	N	
	CI		For Analyses Reques	ted:			
Coolant Type:	TC		Correct Type &	Preservation?	Y	N	
Gel Ice	0		Adequate Volum	ne?	Y	Ν	
Loose Ice		id-Valley					
None	G		Within Hold Time Volatiles:	e?	Y	Ν	
Packing Material:		S/Senvoy		and an and a	N/		
Bubble Bags		,	Voas Free of He	eauspace?	У	N	NA
Styrofoam C	12 The second		TB on COC?	not provided	У	Z	NA
Other:	00003		Metals:				
None			HN03 Preserved	d?	Y	Ν	NA
	2						
C ***ESI Clients Only		1994 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 - 1995 -	Army Corp:	Geiger	(ticks/mi	n) [.]	
Temperature Blank:	°c not provided		Temperatures (IR)	C	°C	C	S
All preserved bottle All preserved accor	s checked Y N NA (vaas)			(left) (middle			(air)
Comments:		Projec	t <u>Managers:</u>				e de reference à la construction de



SAMPLE RECORD LOG

	Samplo	T	T	T	T		Find	 					-	
Sample	Sample Location	Date	Time	Matrix	Donth (ft)	Complete	Field	 	Analyses &	Preservative			Sample Type	MS/MSD
Identification	(LOCID)	Date	Time	Watrix	Depth (ft)	Sampler	Screening						Sample Type (Project or Duplicate)	Collected
	(LOCID)						(ppm)	 					Duplicate)	Conected
								 	1					

SAMPLE RECORD LOG									

SAMPLE RECORD LOG

COC #	Sample Shipping Date	Comments
	Bute	

 	 S	AMPLE RECORD LOG

Cooler ID No.

TAL Work Order _____

COOLER RECEIPT FORM

Proje	ect		
Cool	er received on and opened on by	1	
Temp	(signature) oerature upon receipt: Cooler oC. Temp. Blank oC.	_	
1.	Were custody seals on outside of cooler and intact? a. If yes, how many and where: b. Were signature and date correct?	YES	NO
2.	Were custody papers taped to lid inside cooler?	YES	NO
3.	Were custody papers properly filled out(ink, signed, etc)?	YES	NO
4.	Did you sign custody papers in the appropriate place?	YES	NO
5.	Did you attach shipper's packing slip to this form?	YES	NO
6.	What kind of packing material was used?	-	
7.	Was sufficient ice used?	YES	NO
8.	Were all bottles sealed in separate plastic bags?	YES	NO
9.	Did all bottles arrive in good condition (unbroken)?	YES	NO
10.	Were all bottle labels complete (no., date, signed, pres, etc)?	YES	NO
11.	Did all bottle labels and tags agree with custody papers?	YES	NO
12.	Were correct bottles used for the test indicated?	YES	NO
13.	If present, were voa vials checked for absence of airbubbles and noted if found?	YES	NO
14.	Adequate volume of voa vials received per sample?	YES	NO
15.	Was sufficient amount of sample sent in each bottle?	YES	NO
16.	Were correct preservatives used?	YES	NO
17.	Were extra labels added to pre-tared containers?	YES	NO
18.	Corrective action taken, if necessary: a. Name of person contacted:		

b. Date:

Tacoma

5755 8th Street East

Chain of Custody Record



THE LEADER IN ENVIRONMENTAL TESTING

Tacoma,	WA	98424		
phone 24	3 02	2 2310	fax 252	01

phone 253.922.2310 fax 253.922.5047																					,	TestAmerica Laboratories, Inc.
Client Contact	Project Ma	anager:				Sit	te Co	ontact:						Date	e:							COC No:
Your Company Name here	Tel/Fax:					_		ontact						Carr							Ŧ	of COCs
Address		Analysis T	Turnaround	Time		\mathbf{T}		T	T	T		T	-		T	T	—				ŧ	01 COCS
City/State/Zip	Calenda	r(C) or W	/ork Days (W	/)						17									11			
(xxx) xxx-xxxx Phone	T.	AT if different f	from Below							17									11			
(xxx) xxx-xxxx FAX Project Name:		7	2 weeks							17									1		5	SDG No.
Project Name:		,	1 week							11	11								1			
Site:			2 days							17												
P O #			1 day			mple				11									1			
Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.	Filtered Sa																Sample Specific Notes:
		!				П	\square	-	1		\square	1	\square	\square	_	\bot		\Box				
	───	──′				H	\vdash	+	+	\downarrow	\vdash	+	+		+	+	+	\square	$ \rightarrow $	+	+	
	+	'				H	\vdash	+	+	+	\vdash	+	+	\vdash	+	+	+	\square	\dashv	+	+	1
	+					H	\vdash	+	+-	+ +	\vdash	+	+	\vdash	+	+	+	\vdash	-+	+	+	
						H	\vdash	+	+	+	\vdash	+	+	\vdash	+	+	+	\vdash	+	+	+	
						Η	\rightarrow	+	+	++	\vdash	+	+	\vdash	+	+	+	\vdash	+	+	+	
			'		'	H	+	+	+	++	\vdash	+	+-1	\vdash	+	+	+	\vdash	+	+	+	
			'		'	H	+	+	+	++	\vdash	+	++	\vdash	+	+	+	\vdash	-	+	+	
			'		'	H	-	+	+	++	\vdash	+	++	\vdash	+	+	+	\vdash	+	+	+	
				<u>├</u> ──┤		H	+	+	+	++	\vdash	+	++	\vdash	+	+	+	\vdash	+	+	+	
				 +		H	+	+	+	++	+	+	++	\vdash	+	+	++	\vdash	+	+	+	
Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaO)H; 6= Othe	r		<u> </u>		4	+	+	+	++	+	+	++	\vdash	+	+	++	\vdash	+	+	+	
Possible Hazard Identification							Sam	ple D	Jispc	sal ((A fe	e ma	ay be	asse	ssed	lifs	ampl	es al	re re	taine	ed I	longer than 1 month)
Non-Hazard Flammable Skin Irritant Special Instructions/QC Requirements & Comments: Skin Irritant	Poison B	, 🗆	Unknown			\square		Ret	urn T	To Cl	ient			Dispo	sal E	3y La	b			Archiv	ve F	For Months
Relinquished by:	Company:			Date/Tim	ne:	F	Recei	ived by	y:						Co	ompai	ny:				D	Date/Time:
Relinquished by:	Company:			Date/Tim	ne:	F	Recei	ived by	y:						Co	ompai	ny:			_	D	Date/Time:
Relinquished by:	Company:			Date/Tim	ne:	F	Recei	ived by	y:						Cc	ompar	ny:				D	Date/Time:



WELL DEVELOPMENT FORM

Project:

Well No.:

Date: _____

Personnel:

Development Method _____

Recovery Temp ℃ Depth to Gallons Turbidity Recovery Time pН E.C. Rate Observations Water (ft.) Removed (Ntu) Rate gpm Inches/min

Total Gallons Removed

APPENDIX E

Glossary

GLOSSARY

Accuracy – The agreement between an analytical result and the true value.

Action Limit – A value for results of a quality control analysis that requires appropriate action to be taken to correct the performance of a system or a method that is not in control. Action limits and appropriate corrective actions are specified contractually. Data obtained when a system or method is not in control may be omitted from a regional database. Note: In a multianalyte method, failure to meet the calibration requirement for a small percentage of analytes should not be cause to omit the entire analysis for a sample from the database. Omission should be determined on an analyte-by-analyte basis. Action limits and appropriate corrective actions are specified contractually.

Analyte – That which is identified and quantified in the process of analyzing the sample.

Assessment – The evaluation process used to measure the performance or compliance of sampling and analysis activities.

Audit - A systematic and independent examination to determine whether sampling and analysis activities and related results comply with planned practices, whether these practices are implemented effectively, and whether the nature and extent of these practices are suitable for the sampling and analysis activities they support.

Batch – The number of samples that are prepared or analyzed with associated laboratory quality control samples at one time. A typical batch size is 20 samples.

Bias – The systematic or persistent distortion of a measurement process that causes errors in one direction.

Blank-corrected Result – Refers to an analytical result that has been corrected (mathematically or through analytical procedures) for the contribution of the method blank. The method blank should be processed concurrently. Any correction should account mathematically for all relevant weights, volumes, dilutions, and other similar sample processing elements.

Calibration – The determination of the relationship between analytical response and concentration (or mass) of the analyte.

Certified Reference Material – A reference material accompanied by, or traceable to, a certificate stating the concentration of chemicals contained in the material. The certificate is issued by an organization, public or private, that routinely certifies such material (e.g., National Research Council of Canada [NRCC], Ottawa).

Chain-of-Custody – An unbroken trail of accountability that ensures the physical security of samples, data, and records.

Check Standard – A quality control sample prepared independently of calibration standards, analyzed exactly like the samples, and used to estimate analytical precision and indicate bias due to calibration.

Coefficient of Variation – The standard deviation expressed as a percentage of the mean. Also termed relative standard deviation.

1

Comparability – An indication of the confidence with which one data set can be compared to another.

Completeness – A measure of the amount of valid data obtained from sampling and analysis activities compared to the amount that was expected to be obtained.

Control Limit(s) – A value or range of values against which results of quality control sample analyses are compared in order to determine whether the performance of a system or method is acceptable. Control limits are typically statistically derived. When quality control results exceed established control limits, appropriate corrective action should be taken to adjust the performance of the system or method.

Data Quality Objectives – Data quality objectives are qualitative and quantitative statements that define the appropriate type and quality of data needed to support the objective of a given project.

Detection Limit – In analytical chemistry, a threshold concentration for a compound below which its presence cannot be measured. The threshold concentration results from a number of different influences, including interference from other compounds in the sample or the inherent limits of the measuring instrument in resolving the measurement signal.

Duplicate Analysis – Analysis performed on a second subsample in the same manner as the initial analysis, used to provide an indication of measurement precision.

Field Blank – A simulated sample (usually consisting of laboratory pure water) that is taken through all phases of sample collection and analysis. Results of field blank analyses are used to assess the positive contribution from sample collection and analysis procedures to the final result.

Field Sampling Plan – A plan that includes information about sampling frequency, sampling locations, sampling procedures, chain-of-custody, acceptance criteria, analytical methods, and data quality management.

Guideline – A recommended practice that is nonmandatory.

Interference Check Sample – A sample run by inductively coupled plasma methodology to verify instrument and background correction factors.

Limit of Detection – The lowest concentration of a substance that can be detected with 99% confidence that the substance is positively identified.

Limit of Quantitation – The lowest concentration of a substance that produces a quantitative result.

Matrix – The sample material in which the analytes of interest are found (e.g., water, sediment, tissue).

Matrix Spike – A quality control sample created by adding known amounts of analytes of interest to an actual sample, usually before extraction or digestion. The matrix spike is analyzed using the normal analytical procedures. The result is then corrected for the analyte concentration determined in the unspiked sample, and expressed as a percent recovery. This provides an indication of the sample matrix effect on the recovery of target analytes.

2

Method - A body of procedures and techniques for performing an activity that is systematically presented in the order in which they are to be executed.

Method Blank – A quality control sample intended to determine the response at zero concentration of analyte and assess the positive contribution from sample analysis procedures to the final result. A clean matrix (generally water) known to be free of target analytes that is processed through the analytical procedure in the same manner as associated samples.

Method Detection Limit – The minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero; determined from analysis of a sample in a given matrix containing the element.

Normalize – Perform a data calculation in order to express results in terms of a reference parameter or characteristic.

Percent Relative Standard Deviation – Calculated by dividing the standard deviation by the mean and multiplying by 100.

Precision – The statistical agreement among independent measurements determined from repeated applications of a method under specified conditions. Usually expressed as relative percent difference, relative standard deviation, or coefficient of variation.

Qualified Data – Data to which data qualifiers have been assigned. Data qualifiers provide an indication that a performance specification in the qualified sample or an associated quality control sample was not met.

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 of the type and quality needed and expected by the customer.

Quality Assurance Project Plan – A formal planning document describing the necessary quality assurance, quality control, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

Quality Control – The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Quality control is an element of quality assurance. Quality control samples and auditing/assessment are common quality control activities.

Quantification – The process of calculating the value of an analyte in a particular sample.

Quantification Limit Check Sample – A check sample containing target analytes at concentrations at or near the quantification limit; used to verify routine method performance at the quantification limit.

Recovery – The percentage difference between two measurements, before and after spiking, relative to the concentration spiked, or the percentage difference between a measured value and a true value, as in the case of a reference material or check standard.

Reference Material – A material of known analyte composition that can be used for comparison of analytical results. The reported analyte concentrations have not been certified.

Relative Percent Difference – Difference of two measurements xt and x2 divided by the mean of the measurements, multiplied by 100.

Representativeness – A measure of the degree to which data accurately and precisely represent an environmental characteristic or condition.

Reproducibility – The ability to produce the same results for a measurement. Often measured by determining the relative percent difference, relative standard deviation, or coefficient of variation for an analysis.

Spike – The addition of a known amount of a substance to a sample or a blank.

Spiked Method Blank - See Check Standard.

Standard – A substance of material, the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property of a sample. In chemical measurements, standard often describes a solution of analytes used to calibrate an instrument.

Standard Reference Material – A material with known properties produced and distributed by the U.S. National Institute of Standards and Technology.

Surrogate Spike Compound – A compound that has characteristics similar to that of a compound of interest, is not expected to be found in environmental samples, and is added to a sample before extraction. The surrogate compound can be used to estimate the recovery of chemicals in the sample.

Target Analytes (or Target Compounds) – One or more elements or compounds that are intended to be determined by an analytical procedure (in contrast to tentatively identified compounds).

Validation – Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Can refer to a process whereby environmental data are determined by an independent entity to be complete and final (i.e., subject to no further change), and to have their value for the intended use described by both qualitative and quantitative statements.

Volatilization – The process of vaporizing at a relatively low temperature.

APPENDIX F

Product User Manuals (Provided on CD)

APPENDIX G

Laboratory Control Limits

(Note: Not all control limit information is provided. The expanded Excel spreadsheet is found in the "Native Files" folder of the CD)

Analysis Group Description	Method Description	Method Code											
Soil - 5 day TAT	AK 102/AK103 (DRO	AK102_103	8										
	Ultrasonic Extraction	3550B	1										
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery-	LCS RPD%	MS Recovery	MS Recovery	MS RPD	Surrogate Recovery-	Recover
	o-Terphenyl	84-15-1	3	1	mg/Kg	LOW %	Hiah %		Low %	Hiah %	in the second	Low %	High %
	n-Triacontane-d62	93952-07-9	3	1	mg/Kg				-			60	120
	DRO (nC10- <nc25)< td=""><td>STL00258</td><td>20</td><td>2.3</td><td>mg/Kg</td><td>75</td><td>125</td><td>20</td><td>72</td><td>128</td><td>20</td><td></td><td></td></nc25)<>	STL00258	20	2.3	mg/Kg	75	125	20	72	128	20		
	RRO (nC25-nC36)	STL00383	50	11	mg/Kg	60	120	20	53	116	21		
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery High %	LCS RPD- %	MS Recovery Low %	MS Recovery High %	MS RPD- %	Surrogate Recovery- Low %	
	o-Terphenyl	84-15-1	3	1	mg/Kg	20.000	en straffent fin og en vik ge				1. C. J. C. L. L. L. B. B. B.	60	120
	n-Triacontane-d62	93952-07-9	3	1	mg/Kg							60	120
	DRO (nC10- <nc25)< td=""><td>STL00258</td><td>20</td><td>2.3</td><td>mg/Kg</td><td>75</td><td>125</td><td>20</td><td>72</td><td>128</td><td>20</td><td></td><td></td></nc25)<>	STL00258	20	2.3	mg/Kg	75	125	20	72	128	20		
	RRO (nC25-nC36)	STL00383	50	11	mg/Kg	60	120	20	53		21		
Soil - 5 day TAT	Metals (ICP/MS)	6020											
	Preparation, Metals	3050B	1										
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery- High %	LCS RPD- %	MS Recovery Low %	MS Recovery High %	MS RPD- %	Surrogate Recovery- Low %	Surrogate Recovery High %
	Arsenic	7440-38-2	0.2	0.00078	mg/Kg	80	120	35	75	125	35	Maltin and state	
	Barium	7440-39-3	0.2	0.0015	mg/Kg	80	120	35	75	125	35		
	Cadmium	7440-43-9	0.2	0.00047	mg/Kg	80	120	35	75	125	35		
	Chromium	7440-47-3	0.2	0.004	mg/Kg	80	120		75		35		
	Lead	7439-92-1	0.2	0.001	mg/Kg	80	120				35		
	Selenium	7782-49-2	0.5	0.00188	mg/Kg						35		
	Silver	7440-22-4	0.2	0.00041	mg/Kg						35		

Soil - 5 day TAT Mercury (CVAA) 7471A

Preparation, Mercury	7471A_Prep											
Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	Recovery	LCS RPD- %	MS Recovery Low %	MS Recovery High %	MS RPD- %	States and the states of the	Surrogate Recovery- High %
Mercury	7439-97-6	0.02	0.0063	mg/Kg	75	125	25	75	125	35		

Soil - 5 day TAT	Metals (ICP/MS)	6020]										
	Preparation, Metals	3050B											
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery- High %	LCS RPD- %	MS Recovery Low %	MS Recovery High %	IMS RPD-	C. C	Surrogate Recovery- High %
	Arsenic	7440-38-2	0.2	0.00078	mg/Kg	80	120	35	75	125	35		

Soil - 5 day TAT	PCBs by GC	8082											
	Ultrasonic Extraction	3550B	1										
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery High %	LCS RPD %	MS Recovery Low %	MS Recovery High %	MS RPD- %	Surrogate Recovery- Low %	
	Tetrachloro-m-xylene	877-09-8	0.01	0.1	mg/Kg							45	155
	DCB Decachlorobiphe	2051-24-3	0.01	0.1	mg/Kg							60	125
	PCB-1016	12674-11-2	0.01	0.0032	mg/Kg	40	140	20	40	140	20		
	PCB-1016 Peak 1	STL00132	0.01	0.0032	mg/Kg								
	PCB-1016 Peak 2	STL00290	0.01	0.0032	mg/Kg								
	PCB-1016 Peak 3	STL00232	0.01	0.0032	mg/Kg								
	PCB-1016 Peak 4	STL00307	0.01	0.0032	mg/Kg								
	PCB-1016 Peak 5	STL00085	0.01	0.0032	mg/Kg								
	PCB-1221	11104-28-2	0.01	0.008	mg/Kg							-	
	PCB-1221 Peak 1	STL00206	0.01	0.008	mg/Kg								
	PCB-1221 Peak 2	STL00174	0.01	0.008	mg/Kg								
	PCB-1221 Peak 3	STL00313	0.01	0.008	mg/Kg	1				×			
	PCB-1232	11141-16-5	0.01	0.007	mg/Kg	50	150	30	50	150	30		
	PCB-1232 Peak 1	STL00280	0.01	0.007	mg/Kg	1							

PCB-1232 Peak 2	STL00210	0.01	0.007	mg/Kg	T					1	Т	T
PCB-1232 Peak 3	STL00110	0.01	0.007	mg/Kg								
PCB-1232 Peak 4	STL00108	0.01	0.007	mg/Kg								
PCB-1232 Peak 5	STL00130	0.01	0.007	mg/Kg			-					
PCB-1242	53469-21-9	0.01	0.0021	mg/Kg	57	128	20	57	128	20		
PCB-1242 Peak 1	STL00059	0.01	0.0021	mg/Kg								
PCB-1242 Peak 2	STL00276	0.01	0.0021	mg/Kg								
PCB-1242 Peak 3	STL00020	0.01	0.0021	mg/Kg								
PCB-1242 Peak 4	STL00075	0.01	0.0021	mg/Kg								
PCB-1242 Peak 5	STL00148	0.01	0.0021	mg/Kg								
PCB-1248	12672-29-6	0.01	0.0013	mg/Kg								
PCB-1248 Peak 1	STL00039	0.01	0.0013	mg/Kg								
PCB-1248 Peak 2	STL00268	0.01	0.0013	mg/Kg								
PCB-1248 Peak 3	STL00294	0.01	0.0013	mg/Kg								
PCB-1248 Peak 4	STL00264	0.01	0.0013	mg/Kg								
PCB-1248 Peak 5	STL00218	0.01	0.0013	mg/Kg			-					
PCB-1254	11097-69-1	0.01	0.0021	mg/Kg	65	132	20	65	132	20		
PCB-1254 Peak 1	STL00292	0.01	0.0021	mg/Kg								
PCB-1254 Peak 2	STL00297	0.01	0.0021	mg/Kg								
PCB-1254 Peak 3	STL00022	0.01	0.0021	mg/Kg								
PCB-1254 Peak 4	STL00042	0.01	0.0021	mg/Kg								
PCB-1254 Peak 5	STL00067	0.01	0.0021	mg/Kg								
PCB-1260	11096-82-5	0.01	0.003	mg/Kg	60	130	20	60	130	20		
PCB-1260 Peak 1	STL00060	0.01	0.003	mg/Kg	-							
PCB-1260 Peak 2	STL00274	0.01	0.003	mg/Kg								
PCB-1260 Peak 3	STL00248	0.01	0.003	mg/Kg								
PCB-1260 Peak 4	STL00277	0.01	0.003	mg/Kg								
PCB-1260 Peak 5	STL00197	0.01	0.003	mg/Kg			-					

Water MNA Dissolved Gases (GC RSK_175

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery- High %	LCS RPD %	MS Recovery Low %	MS Recovery High %	MS RPD- %	992665056 2000000 Th	Surrogate Recovery- High %
Methane	74-82-8	5	0.218	ug/L	75	125	20	52	145	20		

Soil - Quick TAT AK 102/AK103 (DRO/ AK102_103

Ultrasonic Extraction	3550B	1										
Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery High %	LCS RPD %	MS Recovery Low %	MSREC - Recovery High	MS RPD- %	Surrogate Recovery- Low %	
o-Terphenyl	84-15-1	3	1	mg/Kg					1000 CT 1000 CT	1700.00	60	120
n-Triacontane-d62	93952-07-9	3	1	mg/Kg	1						60	120
DRO (nC10- <nc25)< td=""><td>STL00258</td><td>20</td><td>2.3</td><td>mg/Kg</td><td>75</td><td>125</td><td>20</td><td>72</td><td>128</td><td>20</td><td></td><td></td></nc25)<>	STL00258	20	2.3	mg/Kg	75	125	20	72	128	20		
RRO (nC25-nC36)	STL00383	50	11	mg/Kg	60	120	20	53	116	21		

	Arsenic	7440-38-2	0.2	0.00078	mg/Kg	80	120	35	75	125	35	And a server how a company	
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCS Recovery- Low %	LCS Recovery- High %	LCS RPD- %	MS Recovery Low %	MSREC - Recovery High	MS RPD- %	Surrogate Recovery- Low %	
	Preparation, Metals	3050B	1										
ick TAT	Metals (ICP/MS)	6020											

Soil - Quick TAT	PCBs by GC	8082]										
	Ultrasonic Extraction	3550B	1										
	Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC Recovery Low %	LCS Recovery- High %	LCS RPD- %		MSREC - Recovery High			Surrogate Recovery- High %
	Tetrachloro-m-xylene	877-09-8	0.01	0.1	mg/Kg				a strand of taken only	A.X. A. C. A. Martin		45	155
	DCB Decachlorobiphe	2051-24-3	0.01	0.1	mg/Kg							60	125
	PCB-1016	12674-11-2	0.01	0.0032	mg/Kg	40	140	20	40	140	20		
	PCB-1016 Peak 1	STL00132	0.01	0.0032	mg/Kg								
	PCB-1016 Peak 2	STL00290	0.01	0.0032	mg/Kg								

PCB-1016 Peak 3	STL00232	0.01	0.0032	mg/Kg	1	1	1		1		
PCB-1016 Peak 4	STL00307	0.01	0.0032	mg/Kg							
PCB-1016 Peak 5	STL00085	0.01	0.0032	mg/Kg							
PCB-1221	11104-28-2	0.01	0.008	mg/Kg							
PCB-1221 Peak 1	STL00206	0.01	0.008	mg/Kg							
PCB-1221 Peak 2	STL00174	0.01	0.008	mg/Kg							
PCB-1221 Peak 3	STL00313	0.01	0.008	mg/Kg							
PCB-1232	11141-16-5	0.01	0.007	mg/Kg	50	150	30	50	150	30	
PCB-1232 Peak 1	STL00280	0.01	0.007	mg/Kg							
PCB-1232 Peak 2	STL00210	0.01	0.007	mg/Kg							
PCB-1232 Peak 3	STL00110	0.01	0.007	mg/Kg			1				
PCB-1232 Peak 4	STL00108	0.01	0.007	mg/Kg							
PCB-1232 Peak 5	STL00130	0.01	0.007	mg/Kg							
PCB-1242	53469-21-9	0.01	0.0021	mg/Kg	57	128	20	57	128	20	
PCB-1242 Peak 1	STL00059	0.01	0.0021	mg/Kg							
PCB-1242 Peak 2	STL00276	0.01	0.0021	mg/Kg			1				
PCB-1242 Peak 3	STL00020	0.01	0.0021	mg/Kg							
PCB-1242 Peak 4	STL00075	0.01	0.0021	mg/Kg							
PCB-1242 Peak 5	STL00148	0.01	0.0021	mg/Kg							
PCB-1248	12672-29-6	0.01	0.0013	mg/Kg							
PCB-1248 Peak 1	STL00039	0.01	0.0013	mg/Kg							
PCB-1248 Peak 2	STL00268	0.01	0.0013	mg/Kg							
PCB-1248 Peak 3	STL00294	0.01	0.0013	mg/Kg							
PCB-1248 Peak 4	STL00264	0.01	0.0013	mg/Kg			1				
PCB-1248 Peak 5	STL00218	0.01	0.0013	mg/Kg							
PCB-1254	11097-69-1	0.01	0.0021	mg/Kg	65	132	20	65	132	20	
PCB-1254 Peak 1	STL00292	0.01	0.0021	mg/Kg							
PCB-1254 Peak 2	STL00297	0.01	0.0021	mg/Kg			1		1	1	1
PCB-1254 Peak 3	STL00022	0.01	0.0021	mg/Kg							
PCB-1254 Peak 4	STL00042	0.01	0.0021	mg/Kg							

PCB-1254 Peak 5	STL00067	0.01	0.0021	mg/Kg							
PCB-1260	11096-82-5	0.01	0.003	mg/Kg	60	130	20	60	130	20	
PCB-1260 Peak 1	STL00060	0.01	0.003	mg/Kg							
PCB-1260 Peak 2	STL00274	0.01	0.003	mg/Kg							
PCB-1260 Peak 3	STL00248	0.01	0.003	mg/Kg				-			
PCB-1260 Peak 4	STL00277	0.01	0.003	mg/Kg							
PCB-1260 Peak 5	STL00197	0.01	0.003	mg/Kg							

APPENDIX H

ADEC Checklist and Technical Memorandum 06-001 (Biogenic Interference and Silica Gel Cleanup)

Laboratory Data Review Checklist

Com	pleted by:			
Title	:			Date:
CS R	Report Name:		-	Report Date:
Cons	sultant Firm:			
Labo	ratory Name:		Laboratory Rep	ort Number:
ADE	C File Number:		ADEC RecKey	Number:
1. <u>I</u>	Laboratory			
	a. Did an A	DEC CS app	roved laboratory receive and perform	<u>n</u> all of the submitted sample analyses?
	C Yes	C No	⊂ NA (Please explain.)	Comments:
	b. If the san laborator	nples were tra y, was the lab	nsferred to another "network" labora oratory performing the analyses AD	tory or sub-contracted to an alternate EC CS approved?
	C Yes	C No	∩NA (Please explain)	Comments:
2 0	hain of Custody (
2. <u>C</u>		10		
	a. COC inform	lation comple	eted, signed, and dated (including rele	eased/received by)?
	C Yes	C No	∩NA (Please explain)	Comments:
	b. Correct ana	lyses requeste	ed?	
	C Yes	C No	C NA (Please explain)	Comments:
3. <u>La</u>	boratory Sample	Receipt Doci	imentation	
	a. Sample/cool	ler temperatur	e documented and within range at re	ceipt $(4^\circ \pm 2^\circ C)$?
	C Yes	C No	○NA (Please explain)	Comments:
[

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

	C No	⊂ NA (Please explain)	Comments:
c. Sample cor	ndition docume	ented - broken, leaking (Methanol),	zero headspace (VOC vials)?
C Yes	C No	⊂ NA (Please explain)	Comments:
d. If there wer preservation,	re any discrepa sample temper	ncies, were they documented? - Fo ature outside of acceptance range,	r example, incorrect sample conta insufficient or missing samples, et
C Yes	C No	CNA (Please explain)	Comments:
e. Data quality	y or usability a	ffected? (Please explain)	Comments:
e Narrative			
ħ.	understandable	e?	
ħ.	understandable C No	CNA (Please explain)	Comments:
a. Present and	C No		Comments:
a. Present and	C No	C NA (Please explain)	Comments: Comments:
 a. Present and C Yes b. Discrepanci C Yes 	C No es, errors or Q C No	 ⊂ NA (Please explain) C failures identified by the lab? ⊂ NA (Please explain) 	
 a. Present and C Yes b. Discrepanci C Yes 	C No	 ⊂ NA (Please explain) C failures identified by the lab? ⊂ NA (Please explain) 	
 a. Present and C Yes b. Discrepanci C Yes c. Were all cor C Yes 	○ No des, errors or Q ○ No Prective actions ○ No	 ∩ NA (Please explain) C failures identified by the lab? ∩ NA (Please explain) documented? 	Comments: Comments:

5. Samples Results

a. Correct analyses performed/reported as requested on COC?

C Yes	C No	C NA (Please explain)	Comments:
b. All applica	ble holding ti	mes met?	
C Yes	⊂ No	C NA (Please explain)	Comments:
c. All soils re	ported on a dr	y weight basis?	
⊂ Yes	C No	⊂ NA (Please explain)	Comments:
d. Are the rep project?	orted PQLs le	ess than the Cleanup Level or the min	nimum required detection level for the
C Yes	C No	⊂ NA (Please explain)	Comments:
e. Data qualit	y or usability a	affected? (Please explain)	Comments:
e. Data qualit <u></u> <u>Samples</u> a. Method Blar		affected? (Please explain)	Comments:
<u>Samples</u> a. Method Blar	ık	affected? (Please explain)	
<u>Samples</u> a. Method Blar	ık		
<u>Samples</u> a. Method Blar i. One me	nk ethod blank re	ported per matrix, analysis and 20 sa	imples?
<u>Samples</u> a. Method Blar i. One me C Yes	ık ethod blank re C No	ported per matrix, analysis and 20 sa	imples?
<u>Samples</u> a. Method Blar i. One me C Yes	ık ethod blank re C No	ported per matrix, analysis and 20 sa	imples?

6.

	C No	∩NA (Please explain)	Comments:
v. Data q	uality or usabi	lity affected? (Please explain)	Comments:
. Laboratory	Control Sam	ole/Duplicate (LCS/LCSD)	
		CCSD reported per matrix, analysis equired per SW846)	and 20 samples? (LCS/LCSD require
C Yes	C No	⊂ NA (Please explain)	Comments:
C Yes	C No	⊂ NA (Please explain)	Comments:
iii. Accura	acy - All perce	nt recoveries (%R) reported and wi	thin method or laboratory limits? An
iii. Accura project sp	acy - All perce ecified DQOs,	nt recoveries (%R) reported and wi if applicable. (AK Petroleum meth 6-120%; all other analyses see the 1	thin method or laboratory limits? An ods: AK101 60%-120%, AK102 aboratory QC pages)
iii. Accura project sp 75%-1259	acy - All perce ecified DQOs, %, AK103 60%	nt recoveries (%R) reported and wi if applicable. (AK Petroleum meth	thin method or laboratory limits? An ods: AK101 60%-120%, AK102
iii. Accura project sp 75%-125% C Yes iv. Precision limits? An	acy - All perce ecified DQOs, %, AK103 60% C No On - All relativ	nt recoveries (%R) reported and wi if applicable. (AK Petroleum meth 6-120%; all other analyses see the h C NA (Please explain) // ve percent differences (RPD) report ified DQOs, if applicable. RPD rep	thin method or laboratory limits? An ods: AK101 60%-120%, AK102 aboratory QC pages)
iii. Accura project sp 75%-125% C Yes iv. Precision limits? And or sample/	acy - All perce ecified DQOs, %, AK103 60% C No On - All relativ	nt recoveries (%R) reported and wi if applicable. (AK Petroleum meth 6-120%; all other analyses see the h C NA (Please explain) // ve percent differences (RPD) report ified DQOs, if applicable. RPD rep	thin method or laboratory limits? An lods: AK101 60%-120%, AK102 aboratory QC pages) Comments: ed and less than method or laboratory orted from LCS/LCSD, MS/DMSD,
iii. Accura project sp 75%-125% C Yes iv. Precisi limits? An or sample/ pages)	acy - All perce ecified DQOs, %, AK103 60% C No on - All relativ d project spec sample duplic	nt recoveries (%R) reported and wi if applicable. (AK Petroleum meth 6-120%; all other analyses see the h C NA (Please explain) // ve percent differences (RPD) report ified DQOs, if applicable. RPD rep ate. (AK Petroleum methods 20%;	thin method or laboratory limits? An lods: AK101 60%-120%, AK102 aboratory QC pages) Comments: ed and less than method or laboratory orted from LCS/LCSD, MS/DMSD, all other analyses see the laboratory of

vi. Do the affected samples(s) have data flags? If so, are the data flags clearly defined?

	C No	⊂ NA (Please explain)	Comments:
vii. Data q	luality or usat	pility affected? (Please explain)	Comments:
c. Surrogates -	- Organics Or	lly	
i. Are surro	ogate recoveri	es reported for organic analyses - fie	eld, OC and laboratory samples?
C Yes	C No	⊂NA (Please explain)	Comments:
project spe the laborat	cy - All percer ecified DQOs, fory report pag	if applicable. (AK Petroleum metho ges)	nin method or laboratory limits? And ods 50-150 %R; all other analyses see Comments:

iii. Do the clearly defi C Yes	sample results ined?	s with failed surrogate recoveries ha	ve data flags? If so, are the data flags Comments:
iv. Data qu	ality or usabil	lity affected? (Use the comment box	to explain.). Comments:
d. Trip Blank - <u>Soil</u> i. One trip b	Volatile anal blank reported er explanation	yses only (GRO, BTEX, Volatile Cl I per matrix, analysis and for each co below.)	Comments: hlorinated Solvents, etc.): <u>Water and</u> ooler containing volatile samples?
d. Trip Blank - <u>Soil</u> i. One trip I (If not, ente	Volatile anal	yses only (GRO, BTEX, Volatile Cl I per matrix, analysis and for each co	Comments: hlorinated Solvents, etc.): <u>Water and</u>
d. Trip Blank - Soil i. One trip I (If not, ente O Yes ii. Is the coo	Volatile anal blank reported er explanation C No oler used to tr	yses only (GRO, BTEX, Volatile Cl I per matrix, analysis and for each co below.)	Comments: hlorinated Solvents, etc.): <u>Water and</u> ooler containing volatile samples? Comments:

	ults less than l	PQL?	
∩ Yes	C No	← NA (Please explain.)	Comments:
iv. If abov	ve PQL, what	samples are affected?	
			Comments:
v. Doto qu	ality or usabil	ity offected? (Discourse 1. i.)	
v. Data qu		ity affected? (Please explain.)	Comments:
e. Field Duplica			
i. One field	duplicate sub	mitted per matrix, analysis and 10 j	project samples?
C Yes	C No	CNA (Please explain)	Comments:
	99799999999999999999999999999999999999		
ii. Submitt	ted blind to lat	o?	
C Yes	C No	○ NA (Please explain.)	Comments:
iii. Precisio (Recom	mended: 30%	The percent differences (RPD) less the water, 50% soil) PD (%) = Absolute Value of: $(R_{4}-I)$	<u>R_2)</u> x 100
	= Sample Cor = Field Duplie	$((R_{1+} R_{2})$)/2)
	C No	C NA (Please explain)	Comments:
C Yes	an a		
	ality or usabili	ity affected? (Use the comment box	to explain why or why not.)

f. Decontamination or Equipment Blank (if applicable)

· · · · · ·	C Yes	C No	C NA (Please explain)	Comments:	
L					
	i. All resul	lts less than PQ	DL?		
r	∩ Yes	C No	CNA (Please explain)	Comments:	
L		DOI 1.			99 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199 - 199
	11. II above	e PQL, what sa	mples are affected?	Comments:	
	iii. Data qu	uality or usabili	ity affected? (Please explain.)		
	iii. Data qu	uality or usabili	ty affected? (Please explain.)	Comments:	
 Other Da			ty affected? (Please explain.) E, AFCEE, Lab Specific, etc.)		
	ata Flags/Q				

Resoutioned

ALASKA DEPARTMENT OF ENVIRONMENTAL CONSERVATION DIVISION OF SPILL PREVENTION AND RESPONSE CONTAMINATED SITES REMEDIATION PROGRAM

Technical Memorandum - 06-001

Date May 18, 2006

Biogenic Interference and Silica Gel Cleanup

PURPOSE:

The Alaska Department of Environmental Conservation (DEC) has developed state specific laboratory methods for the analysis of petroleum hydrocarbons in soil and water matrices, AK101 for Gasoline Range Organics (GRO), AK102 for Diesel Range Organics (DRO) and AK103 for Residual Range Organics (RRO). The methods are located in Appendix D of the Underground Storage Tank (UST) Procedures Manual, November 7, 2002. Additionally, they are adopted by reference in regulation, both in 18 AAC 75 Oil and Other Hazardous Substances Pollution Control, {18 AAC 75.355 (d)}, and in 18 AAC 78 Underground Storage Tanks, (18 AAC 78.007). This technical memorandum addresses the issue of "naturally occurring organic material" (NOM) and/or "biogenic interference" specifically in relation to methods AK102 and AK103. It also provides the laboratory and reporting requirements for utilizing a silica gel cleanup procedure as a method for evaluating the presence of biogenics and thier contribution to the AK102/AK103 sample results.

BACKGROUND:

It is well established that the currently promulgated AK102 and AK103 methods for petroleum range organic analysis are complicated by biogenic interference. NOM or biogenics are present in many soils and especially prevalent in certain Alaskan soils, e.g. tundra peat. As a result, biogenic interference is the term that is used to describe the NOM that is quantified and reported as DRO and/or RRO in accordance with the AK102 and AK103 methods. Biogenic interference concentrations may occur at levels well above regulatory cleanup levels.

Likewise, silica gel cleanup is a well established analytical procedure utilized to separate analytes from interfering compounds of different polarity. The majority of "fresh" or nonbiodegraded petroleum hydrocarbons are considered non-polar compounds. Depending on the soil makeup, the majority of the biogenic compounds may be polar or semi-polar in nature. The silica gel cleanup procedure will preferentially remove polar and semi-polar compound.

In order to ensure consistent data when evaluating the presence and degree of biogenic interference at a contaminated site, the department provides the following laboratory procedure and data reporting requirements.

I. Laboratory Procedure for Silica Gel Cleanup

1. Objective

Alaskan samples containing organic plant material are especially susceptible to background biogenic interference and may result in false positive results for DRO or RRO defined petroleum hydrocarbon ranges. Interpretation of the sample chromatogram MUST be done by an experienced analyst for qualitative match of the chromatograph pattern to known sources of fuel product and/or biogenic interference. Once biogenic interference has been determined, this procedure may be used as an analytical tool to evaluate the contribution of biogenic interference to the original sample results.

2. Method Summary

2.1 A sample extract that has been prepared **and analyzed** utilizing the standard AK102/AK103 methodology, is flushed through a silica gel column using methylene chloride.

Note: The extract must not be acidified. An acid cleanup step is not allowed.

2.2 This silica gel cleanup MUST also be performed on all QC samples in the analytical batch associated with the field sample. At a minimum, this must include the Method Blank (MB), Laboratory Control Sample(s) (LCS) and LCS Duplicate(s) (LCSD). All QC results must be reported with the results of the field samples, before and after cleanup. See QC section of this appendix for required control limits.

3. Apparatus and Materials

- 3.1 Drying oven: an oven capable of maintaining 150°C is used for drying of sodium sulfate and activation/storage of silica gel.
- 3.2 Glassware
 - 3.2.1 Turbo Vap tubes
 - 3.2.2 10mL graduated disposable pipettes or equivalent

3.3 Reagents

- 3.3.1 Methylene chloride analytical grade or better, must be demonstrated to be below method detection limits for diesel and residual range contaminants.
- 3.3.2 Ottawa sand cleaned/baked sand used for soil method blanks.
- 3.3.3 Silica gel Anhydrous, 60 100 mesh. Commercially available prepacked extraction cartridges may be used provided they meet all quality control performance criteria listed in this appendix.

IMPORTANT: silica gel must be activated by placing in a 150°C oven prior to use. Additionally, prolonged exposure to moist air may result in reduced or deficient method performance. Activated silica gel should be stored in a manner as to prevent moisture exposure. It is recommended that the silica gel be stored in the oven continually prior to use.

3.3.4 Glass wool - Pesticide grade or better.

4. Procedure

- 4.1 Preparing the column
 - 4.1.1 Cut the top off a 10mL disposable volumetric Pasteur pipette using a triangular file.
 - 4.1.2 Place a small plug of glass wool into the pipette and slide it down into the taper.
 - 4.1.3 Add a few grams of Ottawa sand to cover the glass wool and provide a flat bed for the silica gel.
 - 4.1.4 Add silica gel to the pipette, with occasional shaking to ensure uniform packing, up to the 3mL mark. Alternatively, the silica gel may be added as a "slurry" with methylene chloride to minimize channeling.
 - 4.1.5 Add another few grams of Ottawa sand to provide some protection to the silica gel bed.
 - 4.1.6 Pre-elute the column with at least 1 volume of methylene chloride.

Note: Overloading of the silica gel column capacity may occur with extracts containing elevated concentrations of biogenics. Dilution or adjustment of the sample extract volume prior to clean up may be necessary to avoid unwanted breakthrough.

- 4.2 Extract Preparation
 - 4.2.1 Fill the column to the ~1.5 ml mark with methylene chloride. Allow methylene chloride to drain down to the sand and discard.
 - 4.2.2 Pipette an aliquot of sample from the vial into the column.
 - 4.2.3 Immediately fill the column with methylene chloride up to the -1.5 ml mark.
 - 4.2.4 When the methylene chloride reaches the 0 ml mark, begin collection of the sample into a 15 ml centrifuge tube.
 - 4.2.5 When the methylene chloride reaches the sand, refill the column to the 1.5 ml mark and continue collecting the sample. Repeat this step twice and allow methylene chloride to drain finish dripping from column.
 - 4.2.6 Using an appropriate concentration device, concentrate extract to the required volume.
- 5. Quality Control
 - 5.1 Calibration requirements and limits are the same as specified in AK102 and AK103.
 - 5.2 QC Results that are outside of the following control limits must be flagged.
 - 5.3 The analysis of a matrix spike and matrix spike duplicate is highly recommended when this procedure is used.

Table 2Acceptance Criteria for QC SamplesAfter Silica Gel Cleanup

4

	Control Limits	
DRO Recovery:	% Recovery	Relative % Difference
Lab Control Samples & Duplicates	70-125	20
Surrogate Recovery:		
Quality Control Sample	70-125	
Field Sample	50-150	

II. Data Reporting Requirements

The department requires the following data to be submitted for the evaluation of biogenic interference in AK102 DRO and AK103 RRO sample results.

- 1. Comparison samples.
 - a. In general, three to five comparison samples must be collected from similar depth and soil strata. The exact number of comparison samples will be determined by the project data quality objectives. The samples will be analyzed for Total Organic Carbon (TOC) and AK102/AK103, before and after silica gel cleanup. The sample results will be utilized for chromatographic interpretation only, to establish the presence of biogenic interference at the site, the general biogenic chromatographic fingerprint and the effectiveness of the silica gel cleanup procedure. The results will not be used to establish a "background concentration" or "average concentration."

2. Analytical Data

- a. The samples must be extracted only once, per methods AK102/AK103
- b. Standard AK102/AK103 sample analysis must be performed and reported
- c. Silica gel <u>column</u> cleanup procedure must performed on the same extract *Note – A silica gel "slurry" or "swirl" is not acceptable
- d. The silica gel cleanup procedure must be performed and reported for all associated quality control (QC) samples
 - i. Minimum required Method Blank, LCS and LCS Duplicate
- e. QC and Surrogate results must be within method specified limits for both analyses (before and after silica gel cleanup).
- f. All sample results and chromatograms and a narrative report must be submitted to the department for evaluation.
- 3. Evaluation
 - a. Results of samples analyzed using the silica gel cleanup procedure will not be accepted as representative of a site without completion of the comparison process described above.
 - b. The presence of biogenic interference must be clearly demonstrated in the background samples. The site samples should contain chromatographically similar biogenic fingerprints. The department will not accept silica gel cleanup results for samples that do not exhibit biogenics and/or only chromatographically exhibit petroleum hydrocarbon contamination.
 - c. If the weight of evidence supports the presence of biogenic interference in the AK102 and AK103 sample results, the department may agree to utilize the silica gel cleanup results for site decision purposes.